# ANNUAL REPORT
# 1985-86

NATIONAL
METALLURGICAL
LABORATORY
JAMSHEDPUR
(INDIA)

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## 1985-86

**NATIONAL METALLURGICAL LABORATORY**
(COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH)
JAMSHEDPUR 831 007

*Edited by:*

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## FOREWORD

I have great pleasure in presenting the Annual Report of the National Metallurgical Laboratory for the year 1985-86.

During this year, the laboratory achieved a significant rise in the recognization and utilization of NML technology and expertise.

Based on the flow-sheet developed by the laboratory, a 72-tonne per day fluorspar beneficiation plant was commissioned at Chandidongri in Madhya Pradesh by MPMMC Ltd. The consultancy was provided for the development of technical flow-sheet, equipment specifications, selection and layout installation of the plant.

Studies on the beneficiation of two magnetite samples from Bihar State Mineral Development Corporation Ltd. were completed and concentrate assaying 85-95 per cent magnetite was produced.

Two samples of chromite from M/s. Mishrilall Mines, Sarubil, were evaluated, and the work on this high grade ore was completed.

Floatation studies on 2 coal fines samples from M/s. CEMPDIL, Bargolai, Assam indicated that concentrate assaying 1.8 and 3.08 per cent ash could be produced.

Bench and pilot plant sintering studies on blue dust samples from M/s. NMDC Ltd., Hyderabad indicated that blue dust to the extent of 22.25 per cent could be successfully used for the production of good quality fluxed sinters.

During that year, M/s. Engineers India Ltd. (EIL) were involved in the evaluation of data generated at the NML Pilot Plants for the production of electrolytic manganese dioxide and manganese metal using ores from different parts of India for preparing a basic engineering package of the technologies. In the feasibility report, M/s. EIL have stated that the process for the exploitation of manganese ores is technically viable.

Ammonia leaching and washing of polymetallic sea nodules at a pH 9.0 carried out during the year indicated high recoveries of over 90 per cent nickel and cobalt. The recovery of copper was found to be 60 per cent and efforts are being made to improve upon the recovery rate.

The copper content of the discarded slag at M/s. Hindustan Copper Ltd., Ghatsila was found to be high and efforts are being made to reduce the same by improving upon the holding process.

A chemical process for the production of pure magnesium carbonate from magnesite, suitable for production of high grade basic refractories was developed. A preliminary techno-economic study of the various processes indicates that the ammonium chloride process is definitely superior to the other processes. Optimum conditions for leaching of calcined magnesite in ammonium chloride solution were established.

By successful cycles of operation, production of electrolytic and activated manganese dioxide (EMD) simultaneously from a single flow sheet has been achieved.

Chemical processing of oils of vegetable origin to upgrade it for use as mould/core binders in foundry, was taken up.

Samples of (*i*) Bolting steels having three different compositions from M/s. Mahindra Ugine Ltd., Khapoli, and (*ii*) Super-heater boiling tube steel, from M/s. Alloy Steel Plant, Durgapur were put to rigorous high temperature creep test programmes for period extending up to 33,000 hours. The analysis of the data generated demonstrate that these creep resistant steels, which are used by thermal power plants, are qualitatively comparable with those produced by developed countries elsewhere.

Fatigue properties of over 150 chain links were studied in details. The chain links are produced by M/s. MAMC, Durgapur, for export to USSR.

Joint collaborative project between NML and Fertilizer (Planning and Development) India Ltd., Sindri, on development and performance evaluation of diffusion treated steels in fertiliser

and chemical industries was successfully completed. The PDIL Sindri contacted 93 manufacturers of sulphuric acid both in Public and Private Sector, informing the success of Aluminium Diffusion Treated Steel products for heat exchanger tube application. A Bombay firm has evinced interest in commercial production of the product.

Creep evaluation tests (short and long term) of boiler quality plates have been carried out to promote substitution of the corresponding imported grades.

Studies were carried out on magnesite dust received from M/s. Almora Magnesite Ltd. Seven binders were used in briquetting with optimum pressure at 750 Kg/Cm$^2$. The properties studied were shatter index, bulk density, porosity, DTA and sintering at high temperature. The results indicated suitability of two of the binders.

In order to develop refractories using beneficiated kyanite and beach sand sillimanite cylindrical sample specimens and bars were fabricated with two grades of sillimanite beach sand using clay as binder and some sintering agents as additives using 15 tonnes pressure. The physical properties such as bulk density, CCS, and bending strength were determined. The samples were further fired in electric furnace to a temperature of 1500°C with 2 hours soaking.

Trials carried out for evaluation of the use of pre-reduced iron in LD converter indicate 100 per cent replacement of scrap by pre-reduced iron with no abnormality in blowing behaviour or any deleterious effect on metallurgical parameters. The average dephosphorisation was better—75 per cent compared to 60 per cent of normal heats and within low phosphorous range of 0.01 to 0.02 per cent. The total blowing time (22.8 minutes against 24.5 minutes of normal heats) and early slag formation improved while lime consumption remained the same (129.2 Kg/tonne of crude steel).

Compacted graphite iron has been produced in direct electric arc furnace, using sponge iron and pig iron as separate base charges. Sponge iron yielded 80 per cent recovery. Melts of sponge after carburization and siliconization was inoculated with conventional nodularisers and denodularisers to produce compacted graphite iron.

Production of tubes with mild steel sandwiched between stainless steel in the inner and outer cases was taken up. The mechanical properties, structure of the inter-faces and nature of bonds formed have been studied for a complete evaluation of such composite tubes of various diameters.

High strength toughness aluminium-lithium alloys with low weight for applications in aircraft are under development.

Shape memory alloys based on Cu-Zn-Al system having varying compositions have been made. Ms temperature and order and disorder transformation temperature of one of the alloy have been evaluated. An alternative heat-treatment procedure has also been devised to avoid the stabilisation effect.

Anisotropic Mn-Al-C magnets by hot extrusion is under development.

The high output aluminium anode (HOPAL) for cathodic protection has been successfully developed and the technology has been transferred to M/s. Dum Dum Valves and Bearings Ltd. For the first time an indigenous design for the cathodic protection of off-shore oil jackets using NML-HOPAL anode and monitoring system has been submitted to the Oil and Natural Gas Commission.

The laboratory has entered into joint collaboration with Oil and Natural Gas Commission. The following projects have been taken up:

1. Development of formulations for transportation of wet sour crude/condensate and gas.
2. Some inhibitors have been synthesised and are being evaluated under simulated laboratory conditions and further development for oil/gas wells acidisation.
3. Development of formulation for water injection system used in oil wells.

Data based on three years exposure tests on Japanese samples both in India and Japan were collected and the reported results were discussed in the last Asian Pacific Corrosion Control Conference held in Tokyo in 1985. The Indian samples are now subjected to exposure in different climatic conditions in Japan. Similar tests with the Indian samples in India have been completed.

*Director*

## SPONSORED INVESTIGATIONS COMPLETED/UNDERWAY

| Sl. No. | Title | Sponsor |
|---|---|---|
| 1. | Determination of some physical characteristics of sponge iron lumps and sponge iron briquettes produced from sponge iron fines | Orissa Sponge Iron Ltd. |
| 2. | Froth flotation studies on Baragolai coal from NEC Assam | CEMPDIL, Ranchi |
| 3. | Mineralogical & locking study of tailings of lead-zinc ore sample from Sargapalli mine project | HZL, Sargapalli |
| 4. | Beneficiation studies on the Salem magnesite samples (bench and pilot plant scale tests) Parts I, II and III (Part I one report, Parts II & III one report) | MDB, New Delhi |
| 5. | Pilot plant scale studies on beneficiation of Bolani and Barsua iron ore slimes | MDB, New Delhi |
| 6. | Sintering studies blue dust sample from Bailadila mines | NMDC Ltd., Hyderabad |
| 7. | Investigation on stainless steel wire rope (two nos.) | Usha Martin Industries Ltd., Ranchi |
| 8. | Metallurgical investigation of the failed mill drive shaft | Patratu Thermal Power Station, Patratu |
| 9. | Investigation on the failure of super heater economiser water wall tubes of Unit No. 3 | Panki Thermal Power Station |
| 10. | Investigation of a crash metallographic defection welded economiser tube | Ennore Thermal Power Station, Tamil Nadu |
| 11. | Failure of water pipe high rise | Tamil Nadu Electricity Board, Tamil Nadu |
| 12. | Investgation on the failure of boiler tube | M.P. State Electricity Board, Korba |
| 13. | Investigation on boiler tube sample of connecting super heater Boiler No. 5 | M.P. State Electricity Board, Korba |
| 14. | Investigation for the determination of alpha quartz and copper in ash samples | M.P. State Electricity Board, Korba |
| 15. | Investigation on failure of Boiler | M.P. State Electricity Board, Korba |
| 16. | Investigation on steam cooled water wall tube | NTPC, New Delhi |
| 17. | Creep evaluation of steel sample | Shivananda Steel Ltd., Madras |
| 18. | High temperature tensile test as per ASTM A-447 type II | Shivananda Steel Ltd., Madras |
| 19. | Investigation on metallurgical examination of dewaxing unit | IOC, Barauni |
| 20. | Investigation on the welded sample of coke chambers | IOC, Gauhati |
| 21. | Testing of 4 Nos. of 50 mm dia tube | IOC Ltd., Haldia |

| Sl. No. | Title | Sponsor |
|---|---|---|
| 22. | Creep tests on the steel samples | Engineers India Ltd., New Delhi |
| 23. | Investigation on the HK 40 failure of reformer tube | Engineers India Ltd., New Delhi |
| 24. | 0.2% proof stress tests on a tube sample at elevated temperatures | Indian Seamless Metal Tube Ltd., Ahmednagar |
| 25. | Hot tensile tests on seamless tubes | Indian Seamless Tubes Ltd., Ahmedabad |
| 26. | Fatigue testing of connecting link (two nos.) | MAMC, Durgapur |
| 27. | Creep rupture testing of AISI 316 austenitic steel specimens | Reactor Research Centre, Kalpakkam |
| 28. | Investigation on failure of microwave tower at Ranchi | P & T, Calcutta |
| 29. | Creep test charges of steel samples | Shama Engine Valves, New Delhi |
| 30. | Charpy impact test | Stewarts & Lloyds (I) Ltd. |
| 31. | Testing of stainless steel wire | Bharat Wire Rope Mfg., Bombay |
| 32. | Metallurgical evaluation of MS plates | Hooghly River Bridge Commission |

## COLLABORATIVE PROJECTS

| Sl. No. | Title | Collaborator |
|---|---|---|
| 1. | Evaluation of pre-reduced iron in LD converter for replacement of scrap | NML-SAIL, RDCIS |
| 2. | Characterisation of boiler quality plates for creep properties | NML-SAIL, RDCIS |
| 3. | Improvement in the manufacturing technique for tar bonded basic refractories | NML-SAIL, RDCIS |
| 4. | Evaluation of clay graphite stopper heads developed by NML (improvements of clay graphite stopper heads) | NML-SAIL, RDCIS |
| 5. | High alumina refractories based on beneficiated kyanite concentrates and beach sand sillimanite dense products & monolithics | NML-SAIL (RDCIS)-CGCRI |

## UTILISATION OF NML TECHNOLOGY

**Processes gone into production (for the first time in 1985-86)**

| *Sl. No.* | *Process* | *Party* |
|---|---|---|
| 1. | Liquid Gold | M/s. Jyoti Refinery, Bombay |
| **Processes released and gone into production (repetitive)** | | |
| 2. | Electric grade alloy aluminium conductor NML PM2 | M/s. Rishi Alloys Pvt. Ltd., Muzaffarnagar |
| 3. | -do- | M/s. Fort Gloster Industries Ltd., Calcutta |
| 4. | Submerged arc welding flux | M/s. Electro-Flux Pvt. Ltd., Hyderabad |

## CONSULTANCY SERVICES RENDERED

| *Sl. No.* | *Title* | *Sponsor* |
|---|---|---|
| 1. | Engineering consultancy services for cathodic protection of off-shore drilling equipment | M/s. Dum Dum Valves & Bearings Pvt. Ltd., Calcutta |
| 2. | Engineering consultancy services for optimum beneficiation approach of Semra magnetite deposit | M/s. Bihar State Mineral Development Corpn., Ranchi |
| 3. | Engineering consultancy for recommending optimum beneficiation approach—Bishrampur graphite | M/s. Bihar State Mineral Development Corpn., Ranchi |
| 4. | Visit and on-the-spot study of graphite and magnetite project sites and advise on the same for their proposed beneficiation plants | M/s. Bihar State Mineral Development Corpn., Ranchi |
| 5. | Engineering consultancy services to examine their kyanite sample from Pardi for preparation/development of technical flow-sheet and advice on the same | Maharashtra Minerals Corpn. Ltd., Bombay |
| 6. | Engineering consultancy services for offering advice on (*a*) preparation of a technical flow-sheet based on the results obtained through tests and studies done at NML on the flourspar sample (*b*) equipment specifications and (*c*) preparation of tender papers and scrutiny of the same for selection of equipment and machinery | M.P. & Maharashtra Minerals & Chemicals Pvt. Ltd., Chinchola, M.P. |

# R & D HIGHLIGHTS

## A. ORE DRESSING & MINERAL BENEFICIATION

During the period 1985-86, R & D work in the fields of mineral beneficiation, agglomeration, utilisation of metallurgical wastes, recovery of bye-products for use in mineral based metallurgical industries including mineralogical, petrological and microscopic studies on ores and minerals was continued.

A short account of the R & D investigations completed as well as those in progress during the above period is given below.

### 1. Pilot Plant studies for development and optimisation of process flow-sheet for magnetite sample from Semra for M/s. Bihar State Mineral Development Corporation, Ranchi

Based on bench scale studies, a tonnage lot of the magnetite was treated in the pilot plant following the optimum route for collecting technical data and finalising the process flow-sheet. Detailed proposal for setting up of a 200 tpd treatment plant for magnetite is being finalised.

### 2. Beneficiation of high silica limestone sample from Gola area for Directorate of Mining and Geology, Patna

The limestone sample assayed 41.07% CaO, 0.38% MgO, 20% insolubles, 2.0% $Al_2O_3$ and 0.61% FeO. Bench scale flotation tests at 38%, $-200$ mesh employing three cleanings gave a concentrate assaying 54.75% CaO with 4.21% insoluble and 83.1% distribution in it. The concentrate with an yield of 63.3% analysed 0.35% $SiO_2$, 0.52% $Al_2O_3+Fe_2O_3$ and 0.39% MgO. This super concentrate would meet most specifications for chemical and metallurgical industries.

### 3. Reduction of silica content in a high silica limestone sample from Damua Naresh-garh area, Bihar for Directorate of Mining and Geology, Patna

Preliminary flotation studies for reducing the silica content have been carried out.

The received sample assayed 44.07% CaO, 1.77% MgO, 0.30% $Fe_2O_3$ and 19.83% insolubles (12.73% $SiO_2$). Froth flotation yielded a refloat concentrate assaying 51.63% CaO with 3.8% insolubles for a CaO recovery of 92% in it which should be acceptable for cement manufacturer. Reflotation of the intermediate floats (Float II & III) obtained from primary flotation would yield a combined product (when mixed with Float I) assaying 51.96% CaO and 3.19% insolubles with 84% recovery.

### 4. Studies for utilisation of low grade tungsten ores of India (1) Wolframite (2) Scheelite

Studies were initiated to develop process to beneficiate Indian tungsten ores (1) Wolframite from Bankura and (2) Scheelite from U.P. Some encouraging results using froth flotation route to produce preconcentrates analysing 15 to 20% $WO_3$ were achieved.

### 5. Development and optimisation of technical flow-sheet for graphite beneficiation plant (Bishrampur sample) for M/s. Bihar Development Corporation, Ranchi

Bench scale beneficiation studies indicated that the graphite sample from Bishrampur area would yield a graphite concentrate assaying 92.25% fixed carbon with 3.56% ash from a feed assaying 9.26% F.C. with 88.64% ash. Pilot plant scale studies are being planned to collect more technical data, besides confirmation of the bench scale results for finalising the process flow-sheet for the proposed beneficiation plant.

### 6. Exploratory studies for lowering the silica content in the fluorite samples received from Chandidungri flurite project of M/s. Madhya Pradesh and Maharashtra Mineral Corporation Ltd.

Three samples from the operating plant corresponding to the current feed and two products were studied, for review of their response to the NML flow-sheet, based on which the plant was set up. The results indicated that by employing the same process and reagents as also with additional cleaning stages it was still possible to produce the desired quality of concentrate though the feed grade deteriorated from 29% to 20.78% $CaF_2$.

### 7. Production of high grade limestone concentrate from high silica limestone sample from Bentibagda area of Bihar

Exploratory studies were undertaken for developing methods for production of limestone super concentrates for use in steel plants. By employing froth flotation route it was possible to produce limestone concentrate assaying as low as 1% silica.

## B. REFRACTORIES TECHNOLOGY

### 1. Improvement in the manufacturing techniques for tar bonded basic refractories—SAIL-CSIR coordinated project

Laboratory scale studies show general improvement in the physical properties of the compositions made with the improved (process) tar as compared with the original tar i.e. without any processing.

Retained carbon which is an important factor in the tar bonded dolomite compositions is about 2.05% with original tar, 2.1% with improved tar and additives in tar increases up to 2.9% which is a significant increase.

### 2. High alumina refractories based on beneficiated kyanite fines and beach sand sillimanite

Seven batch compositions each with the two varieties of sillimanite beach sand Q-grade and M-grade were made with additives and sintering agent. Cylindrical specimens (5cm×5cm) and rectangular bars (15cm×2.5cm×2.5cm) were pressed. The specimens were fired to 1500°, 1550° & 1600°C. The properties like MOR, CCS, bulk density, porosity and PLC were studied. Encouraging results were obtained, in specimens with porosities varying from 12% to 20% were obtained.

### 3. Suitability of Indian sea water magnesia from refractory use

The studies showed that the magnesia produced by CSMCRI, Bhavnagar was high in $B_2O_3$ and fluxes. The total impurities amounted to nearly 12% after high temperature firing. Although a large proportion of $B_2O_3$ volatilised at 1650°C but appreciable amount (0.14%) still remained.

It was concluded that although the material was comparable to some of the standard quality sea water magnesia produced abroad but for wider application the $B_2O_3$ content should be lowered to 0.05%, and CaO to $<3.0\%$ keeping $CaO/SiO_2 > 2$. The total MgO content should thus be raised to 95% (minimum).

### 4. Development of improved ramming mixes for application of steel making furnaces and electrical furnace and secondary refining

Binding characteristics of some binders for use in the basic ramming mixes were studied. Preparations are being made to dead burn one variety of magnesite of Indian resources.

### 5. Development of ceramic recuperators and heat exchangers for high temperature furnaces and kilns

6″×1″×1″ bar specimen from the SiC available in the division were pressed. Five compositions thus formulated were fired at 1200° and 1450°C. Property evaluation of these compositions are in progress for ascertaining the suitability of such a composition for recuperator tube making.

### 6. Evaluation of clay-graphite stopper heads developed by NML (Improvement of clay-graphite stopper heads) SAIL-CSIR Coordinated Project

Service trials of 5 numbers of clay-graphite stopper heads (pin type) developed by NML were carried out at Alloy Steels Plant Durgapur during May, 1985 in the refurnacing ladle of 10-ton electric arc furnace (heat nos. 9-3002, 9-3004, 9-3005, 9-3007 and 9-3008). The trials were successful. For final trials, 15 nos. of clay-graphite stopper heads (pin type) were made and fired under reducing condition at 1450°C for a soaking period of 3 hours in a down draught kiln, and various properties were determined. Final trials are expected to be completed very soon.

### 7. Development of dense high-purity alumina grains from technical alumina

Physico-chemical and refractory properties of some high sintered (1750°C) samples were determined. Maximum bulk density obtained was 3.80 gms/cc. and the alumina content was over 99.2%. The project work is completed on bench scale and the report is under preparation.

### 8. Development of MgO-Carbon refractories

Different batches of MgO with proper gradings were made from 12, 16 and 20% by weight graphite additions. Resins were used as bond. Samples were made at 15 tons of total load and were cured at 120°C for 24, 48 and 72 hours respectively. Properties such as bulk density, apparent porosity, cold crushing strength and modulus of rupture (cold) were determined. A sample of MgO-Carbon refractories received from an indigenous firm was also studied and properties were compared. Further work is in progress.

## C. EXTRACTION & CHEMICAL METALLURGY

### 1. Processing of polymetallic sea nodules for recovery of metallic values

Two independent processes have been developed on Pilot scale based on ammonical leaching reduction-roast, ammonia leaching and direct ammonical leaching of the ground sea nodules. The trials made on reduction-roast ammonical leaching route have been found to be most effective for extraction of copper, nickel and cobalt in comparison to the direct ammonical leaching process. Recoveries of Cu and Ni were above 90% in solution while cobalt was about 80%. Slovent extraction and electrowinning experiments for recovering Cu, Ni and Co were carried out. Manganese was recovered as manganese dioxide from the residue obtained from direct ammonia leaching by acid treatement followed by further processing to EMD.

### 2. Reduction of copper content in dump slag at ICC Ghatsila

The project has been sponsored for a period of 2 years. Smelting trials were carried out on 1 Kg. scale and the results have been found to be encouraging.

### 3. Plating of graphite particles with copper

Technique for depositing copper on graphite particles by electroless process has been developed and the process is ready for exploitation.

### 4. Production of pure magnesium carbonate from magnesite by chemical methods

Extensive work was carried out on the newly developed ammonium chloride process to determine the optimum conditions for leaching of calcined magnesite. Studies for the removal of calcium from the process liquor indicated that the calcium content can be brought down to 0.7 gm CaO/litre.

### 5. Production of electrolytic, activated and chemical manganese dioxide simultaneously from a single flow sheet

It has been experimentally found that the spent electrolyte from electrolytic manganese dioxide (EMD) cells can be regenerated by reacting with calcined manganese ore in successive cycles. This will eliminate reduction of manganese ore presently practised prior to leaching of manganese ores in the spent electrolyte in the production of EMD. The residue obtained amounting to 50-60% of the calcined ore used, is found to be activated for use in dry cells.

### 6. Silver Powder

A method has been developed to produce microfine silver powder by electrolysis in an aqueous solution of suitable salt with standard bullion silver as anode and stainless steel as the cathode. The optimum composition of the electrolyte, pH, cell voltage, current density etc. have been determined. Anodic and cathodic efficiency of 99% and 98.9% respectively have been obtained. The silver powder obtained is of predominantly spherical shape with size in the range of 0.1$\mu$ to 0.3$\mu$. Purity of the silver powder obtained is more than 99.99% with electrical conductivity of 102 in the IACS scale. The hardness, density and other properties of the powder and pellets made out of the powder with and without cadmium oxide have been studied.

### 7. Large scale electrometallurgical facilities

Engineers India Limited, have evaluated the various data collected at NML pilot plant on production of electrolytic manganese dioxide and manganese metal using manganese ores from different parts of India with a view to preparing basic engineering pakage of the technologies. EIL have stated that the process is technologically feasible for exploitation of manganese ores. Further, as desired by EIL, the indirect fired rotary kiln at the electrometallurgical facility has been modified to operate as a direct fired kiln.

### 8. Scale up trials of low temperature (below 500°C) sulphatisation roasting of chalcopyrite concentrates

Detailed analysis of the roasted products of chalcopyrite concentrates obtained in different batches by the larger scale sulphatisation roasting trials of 8.8 kg per batch were carried out.

Both, as received, and chemically treated chalcopyrite concentrates, in −200 mesh particle size, mixed with $R_2O_3$ type additives, gave almost similar results as in 1 kg batch (Lynder type furnace).

Water soluble copper and zinc obtained using as received concentrates were around +94% and +80% respectively, with iron around 1% to that of feed content.

With chemically treated concentrate (zinc and lead 0.05 & 0.02% respectively) water soluble copper and zinc around +98% and +88% respectively with low iron (0.45%) were obtained.

Bulk leaching of the roasted products from as received samples (2 kg per batch) and crystalization of the solution gave pure grade copper sulphate crystals (as per ISI specification) only in the first crop.

Subsequent crops, although conformed to commercial grade with respect to copper contents, were contaminated with zinc in appreciable quantity.

Bulk leaching of the roasted products obtained with chemically treated samples and subsequent crystalization of the solution gave purer grade copper sulphate up to 3rd crop.

Cyclic leaching was not carried out.

## D. IRON & STEEL TECHNOLOGY

### 1. Evaluation of the use of pre-reduced iron in L.D. Converter (SAIL-CSIR Multi Agency Project)

Trials for 100% replacement of scrap by pre-reduced iron (PRI) in 100/130-tonne L.D. converter of Bokaro Steel Plant have been successfully carried out.

It was found that about 63% of the heats finished with a lower opening temperature (1600-1610°C) against the desired target of 1630°C, which is easily remediable.

Phosphorus level in the finished steel was lower, while sulphur in some of the heats was around 0.02%. It was argued that this dual effect was due to two reasons i.e. lower opening temperature and lower basicity of the final slag.

ing in early fluid slag formation.

Operational characteristics related to slopping, spitting or such unusual tendency was absent and the experimental heats compared equally with the normal all scrap heats. Considering a lower specific volume (0.68) of the Bokaro 100/130-tonne L.D. converters, the above facts carry special importance. It may be suggested that the practice could be adopted in all L.D. shops in steel industry in India.

**2. Use of the non-isothermal, thermoanalytical model developed at NML for correlating the actual operating parameters of a Vertical Retort for DRI production with the model, incorporating geometrical mechanical and dynamic similarity criteria**

Under the present situation of deteriorating raw materials in Iron & Steel industry in India, one would have to assess whether inferior but cheaper raw materials, if exploited would lead to more economical operation. Useful information regarding this can be obtained if some standard tests are redesigned so as to take into consideration the following:

(*a*) the interdependence of the various characteratics of a material, and

(*b*) the nature of dependence of a particular property on the operating conditions.

In order to obtain more useful results, a new technique (non-isothermal technique) was developed, and a bench scale experimental set up was designed at NML to obtain a desired end result under varying operating conditions rather than carrying them out under fixed condition to obtain variable end results. The technique serves as a close simulating hot model for vertical retort process, as it employs moving bed. In this study, emphasis has been laid mainly on the assessment of reducibility of iron ore and reactivity of coal under conditions more akin to actual reactor.

The results so obtained are being used in developing a mathematical model correlating the different variables and studying their effect on the production of DRI for the NML's VR/DR process. This would help in assessing the energy conservation, production rate for a particular set of raw materials and also the comparison between the two sets of raw materials.

**3. Agglomeration of DRI fines (sponge iron) with suitable binder for use in melting furnace**

Due to degradation of iron ore during direct reduction processes, a considerable amount of DRI fines are generated. These fines, particularly —3 mm fraction, do not find any direct use in melting and is by and large wasted. Taking the installed DRI capacity in India to be 4,20,000 tonnes per annum (approx.) 60,000 tonnes of fines are generated and this is a colossal waste. A suitable method to agglomerate these fines to make it usable would be a real advantage. Briquettes produced of these fines would find use in melting due to higher specific gravity and bulk density in addition to decreased tendency of rusting and oxidation.

An exploratory work has been undertaken to make strong briquettes by using various binders under different compacting load. The results so far obtained exhibit very strong agglomerates with compression strength $> 3$ tonnes, shatter valves of 93% and tumbler valve of 78%. These are more suitable for melting in EAF.

**4. Physico-chemical Assessment of iron ore/sinter/pellets for their use in blast furnace and direct reduction process**

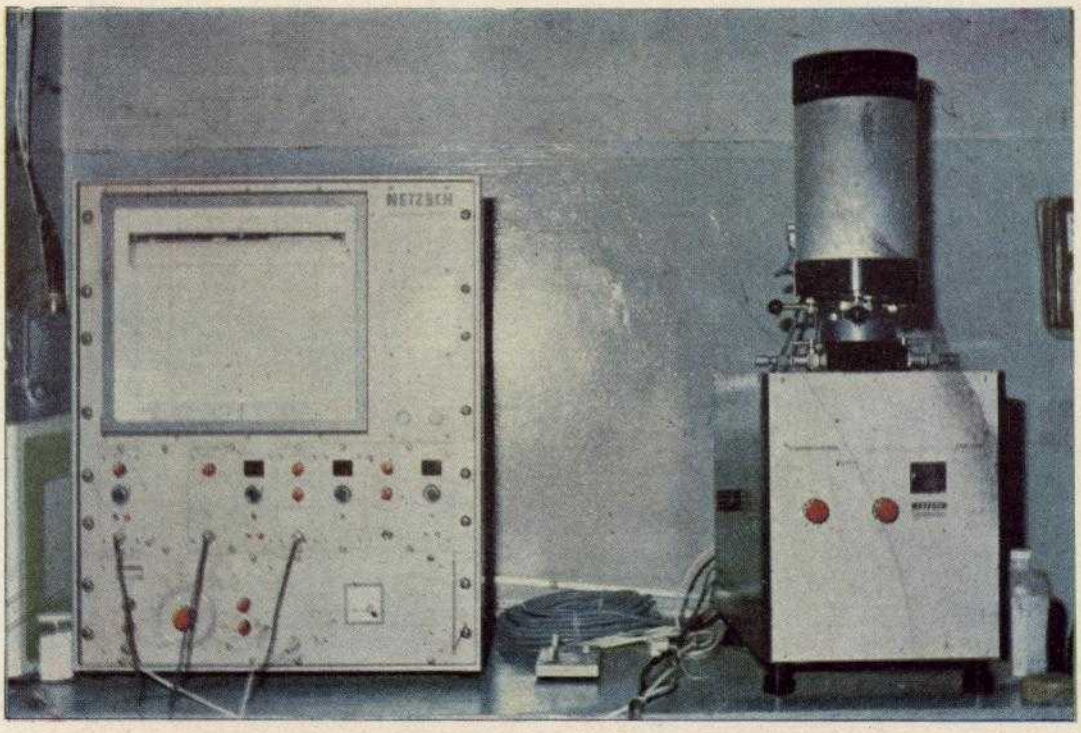
*Completed*

(*a*) Static bed matching test of iron ore with non-coking coal (Western Coal Fields). Sponsor M/s. Bhilai Engineering Co. Ltd.

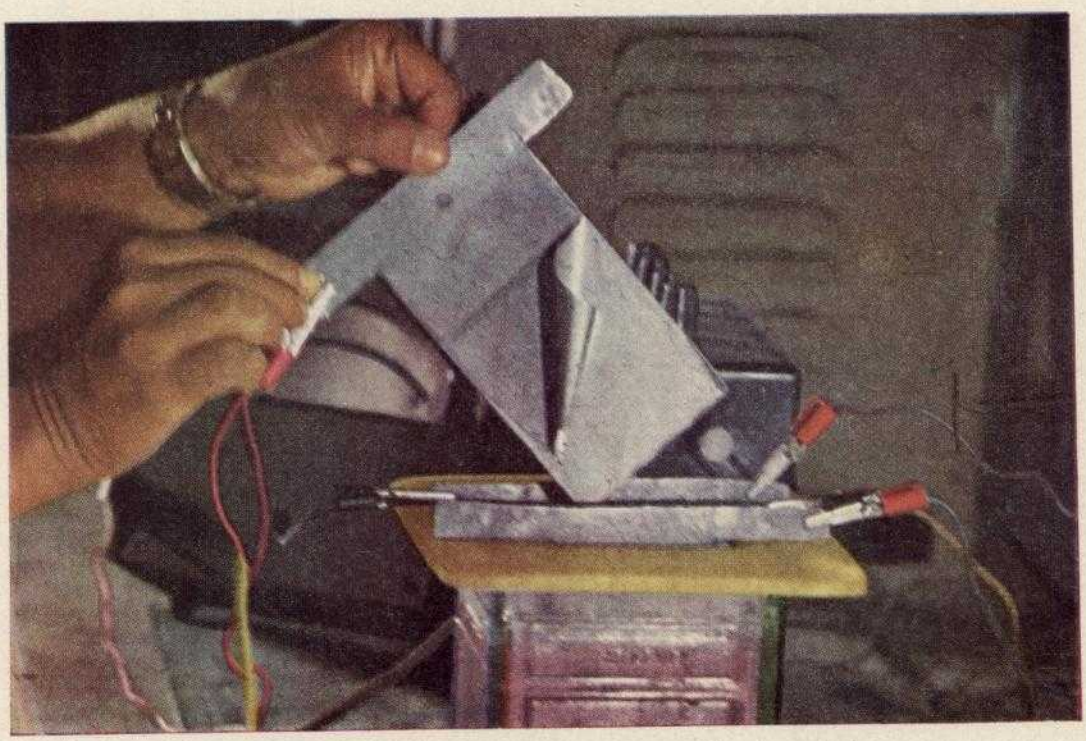
(*b*) Determination of reduction degradation indices of Bhilai Iron Ore/Sinters, 6 samples, Sponsor: M/s. Bhilai Steel Plant Ltd.

*In progress*

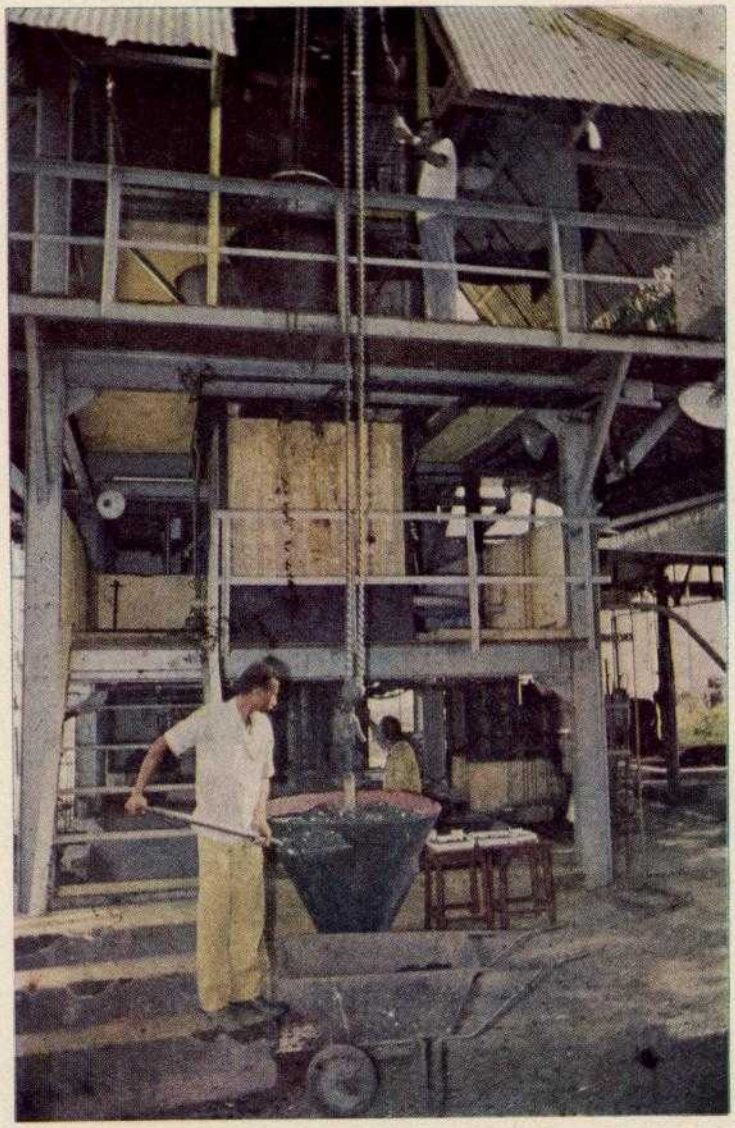
(*a*) Physico-chemical evaluation of cold bonded pellets from RRL Jorhat. This is a collaborative project with SAIL/RRL Jorhat.




*TG/DTA/DTG apparatus for thermal analysis of ceramic materials*



*Electrolytic nickel recovered from polymetallic sea nodules*



*NML's VRDR process for production of sponge iron using non-coking coals*

## E. LIGHT METALS AND ALLOYS

### 1. Transfer of Technology of Aluminium Alloy Conductor NML-PM215

The technology of production of high strength, medium conductivity aluminium alloy conductor, designated NML-PM215, has been transferred successfully after shopfloor demonstrations to M/s. Electrical Manufacturing Co. Ltd., Calcutta in August 1985. The NML-PM215 conductors develop an electrical conductivity of 53% IACS at a tensile strength level of 30 kg/mm$^2$ and conforms to IS: 398 part IV.

*Commercial Production of NML-PM215 alloy conductors*

M/s. EMC, Calcutta is now producing the NML-PM215 alloy conductors on commercial scale.

(*i*) The production of 36 Km length each of overhead conductor (7/3.35 mm) against orders placed by State Electricity Boards of Tamil Nadu and Kerala has been completed.

(*ii*) Against the order of 100 Km length of overhead conductor from Karnataka State Electricity Board, EMC has already despatched 30 Km length conductor.

(*iii*) Against the order of 100 Km length of overhead conductor from Gujarat Electricity Board, EMC had despatched about 40 Km length conductor.

(*iv*) *To study the influence of filtration through NML Reactive Filter on the stress corrosion behaviour of Aluminium Alloys*

Billets (3″ dia) of aluminium alloy NML-PM215 and Al-Zn-Mg-Cu (7075) were cast with and without using ceramic reactive filter. Billets were homogenised and extruded to obtain rods of 1″ dia. C-rings were prepared. Stress corrosion cracking tests of the C-rings were carried out in 3.5% NaCl solution under different pH conditions. Further work is in progress.

### 2. (*i*) Processing of NML-PM215 alloy conductor

M/s. Assam Industrial Corporation, Patna showed interest in NML-PM215 aluminium alloy conductor and desired to go in for production of alloy conductor in their plant. In this connection, a visit was made to the plant for finding the feasibility of processing of NML-PM215 conductor. Further work is in progress.

### (*ii*) Productivity improvement of aluminium utensil small scale Industry

M/s. Sureka Metal Industry, Begusarai, referred the problem of high rejection of aluminium utensils in their plant and desired to implement the NML know-how of improved processing technology for quality and productivity improvement of aluminium utensils. A visit was made to the plant to examine the present practice and infrastructural facilities. Further work is in progress.

### 3. Study on the effect of chlorine and nitrogen on the solidification behaviour of Al-Si and Al-Zn-Mg alloys

Al containing 8, 12 and 16% Si, Al-5Mg and Al-4Zn-2Mg (1 kg each) was prepared employing chlorine and nitrogen gas mixture through a refractory diffuser system which was specially designed with a view to utilising a fraction of quantity of chlorine that is conventionally being used and thereby minimising pollution markedly and at the same time achieving an effective degassing. The system of degassing further enables to disperse uniform fine bubbles of the gas mixture. Density, tensile strength, microstructural details for microporosity and structural refinement were studied and compared with data on the conventionally chlorine degassed simultaneously cast alloys. Improved density, tensile strength, lower porosity counts along with structural refinement in terms of fine Si needles and grain size have been observed with the present arrangement of degassing through the diffuser.

The work was scaled up to 5 kg in the laboratory.

### 4. Solidification behaviour of Al alloy composites

Composite production through this approach of casting from the semi solid state is new. Semi solid state casting enables retention of immiscible phases in a desired distribution pattern in the matrix, as has been experienced in earlier work in this laboratory and these materials showed improved resistance and thermal expansion characteristics as compared to casting from superheated melt. The present work is rather an extension of the earlier work of the laboratory.

An apparatus fabricated in the workshop for this work needed further modification to improve the safety factor. This is being done and the work is expected to start soon. In the mean time a few typical compositions based on Al-Cu-Mn and Al-Zn-Mg have been selected for studies in this set up. Lithium containing alloys also will be studied for their solidification behaviour with this arrangement.

### 5. Preparation of lithium bearing alloys by non-conventional route

A few heats of Al-Zn-Mg containing Li and also Al-Cu-Mn containing Li were recently prepared on a 5 kg scale introducing Li through non-conventional route. The alloys were homogenised for 24 hours and rolled. The work is in progress.

Samples from Al containing Li, prepared earlier were studied through TEM for morphological features and structure of second phase. Intermediate phase with leafy structure and having colonies of eutectic have been observed. The work is in progress to identify the phase.

## F. MAGNETIC MATERIALS

### 1. Anisotrophic Mn-Al-C magnets by hot extrusion

Some structural studies (mostly optical) have been carried out and heat treatments for stabilising the structure have been established. A few trials have been made to get defect free castings of Mn-Al-C (size=75 mm dia, 240 mm ht.). Efforts are now being made to extrude the billet at temps. 600°-700°C, in order to develop anisotropy.

### 2. Materials for portable generators

In order to evolve a suitable magnetic material for portable generator application, magnetic measurements have been carried out from room temperature to about 700°C on 0.4% C-Steel, 4340 type of steel and HSLA steel (containing 0.07% Nb) in quenched, quenched and tempered, and normalised conditions. 4840 type of steel in quenched plus tempered condition and HSLA steel in normalised conditions have exhibited desirable magnetic properties for portable generators. The properties were quite stable up to 400°C. The only draw back is the higher coercive force which may lead to larger losses. The mechanical properties of these materials are expected to be high as desired in portable generator applications and these properties are also being evaluated.

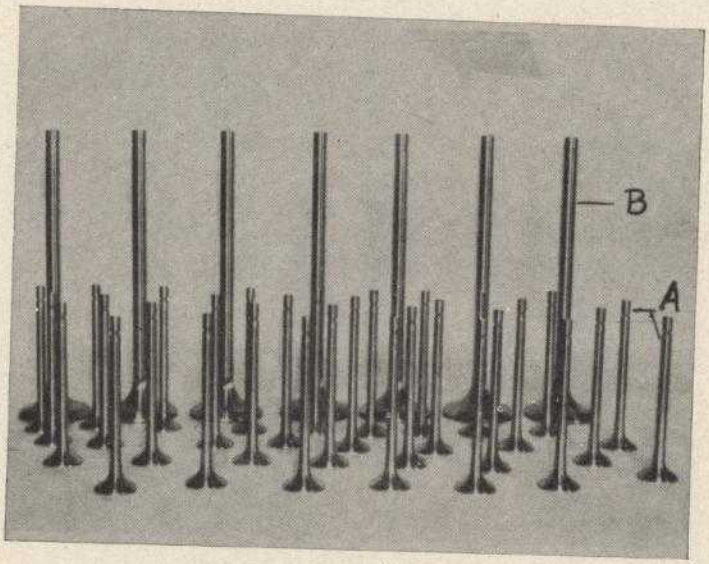
*Low cobalt magnetic alloys*

Low Cobalt Fe-Cr-Co permanent magnets containing 10-12 wt.% cobalt, 28-33 wt.% Cr and balance Fe (with or without small additions of Al, Si, Nb, Cu) have been made through deformation aging technique. The alloys after casting and solution treatment, were spinodally decomposed, deformed (wire drawing, rolling) to get an area reduction of 60% and suitably heat treated. The alloys were studied by TEM and magnetic measurements. The best magnetic properties obtained after a total heat treatment time of 12 h are: remanence=10,600 gauss, coercive force=580 Oe, max. energy product=3.4 MGOe.

## G. HIGH TEMPERATURE CREEP RESISTANT STEELS

### 1. Indigenization of creep resistant steels for thermal power plants

Thermal power generation is heavily dependent on the availability of several grades of creep resistant steels. These steels are required in various shapes, e.g. tubes, pipes, bolts, forgings




*Processed exhaust valves of Cr-Mn-N-C-W-Mo stainless steel*
*(A) As per Tata D1 Engine*
*(B) As per Diesel Locomotive WD $M_2$ Engine*

(weighing up to 75 tonnes) and castings. Till now, power plant equipment manufacturers in India have been mostly dependent on import of such steels. This dependency in the vital power sector of strategic importance motivated the Bharat Heavy Electricals Ltd., Steel makers, statutory bodies like the Central Boiler Board, R & D scientists and engineers to take necessary steps towards indigenization of the internationally known grades of creep quality steels. Obviously, availability of the remarkable steel making capabilities within the country was an encouraging factor.

Systematic beginning in this direction was made in the early 70s with the creation of a voluntary body, the Indian Creep Panel. This facilitated accomplishment of two prerequisite tasks: (1) establishment of a national creep testing facility at NML with the aid of UNDP/UNIDO; and (2) rationalization of important grades of creep resistant steel from amongst the jungle of specifications.

Commercial heats of creep resistant steels were produced by the Alloy Steel Plant (ASP), Durgapur, and the evaluation of short and long term creep behaviour began in 1974 at NML with the ultimate objective of indigenization and import substitution of creep quality steels. The following grades were put through a rigorous high temperature creep evaluation programme for periods extending up to 33,000 hrs i.e. about four years in compliance with ISO practice.

(*i*) *Bolting steels*

(*a*) $1\frac{1}{4}$Cr-1Mo-$\frac{1}{4}$V-Ti-B (GOST 2591)$\frac{1}{4}$
(*b*) $1\frac{1}{4}$Cr-$\frac{3}{4}$Mo (En-20B)
(*c*) 1Cr-1Mo-$\frac{1}{4}$V (DIN 17240)

(*ii*) *Superheater boiler tubing steels*

(*a*) $1\frac{1}{4}$Cr-$\frac{1}{2}$Mo (ASTM 213 T-11)
(*b*) $2\frac{1}{4}$Cr-1Mo (ASTM 213 T-22)

Today prolonged painstaking creep rupture evaluation work carried out at NML has led to the generation of creep rupture data up to 33,000 hrs on all the above steels. The analysis of such data vis-a-vis international/collaborator's data has demonstrated that indigenously produced steels are qualitatively comparable with those produced by advanced countries elsewhere. This in turn has created confidence in the indigenously produced steels and BHEL has already resorted to import substitution in respect of bolting steels as above. Furthermore, certificates have recently been issued by NML in favour of two grades of $2\frac{1}{4}$Cr-1Mo and $1\frac{1}{4}$Cr-$\frac{1}{2}$Mo (both produced by ASP) to the Central Boiler Board for their acceptance.

With the bolting steels already being produced indigenously and with the commencement of production of the two superheater boiler tubing steels by ASP, the anticipated savings of foreign exchange are estimated to be over Rs. 100 million/annum assuming that the entire demand is met by the ASP production.

**2. Multi-agency project (MAP-1): Collaborator NML/SAIL: characterisation of boiler quality plates for creep properties**

Creep properties on the three casts of boiler quality plates have also been generated for a test duration of 3,000 hours at 400°, 450° and 500°C. Optical metallographic studies were completed on the above three casts and a final report is under preparation.

**3. Development of nickel-free creep resistant austenitic steel for automotive exhaust valve application**

Service trial on two exhaust valves fitted in WDM2 Loco No. 18801 at Bondamunda Diesel Shed by Eastern Railway has been successfully completed. Besides, the other two exhaust valves fitted for service trial in Loco No. 18831 at Ratlam Diesel Shed have also successfully completed its duration of one year on 4th April 1985.

RDSO, Lucknow has now shown interest in extensive field trial of the NML valves on 50 Locos i.e. 3200 valves to be made and tried. Steel manufacturers have been approached

for the production of one ton industrial heat of this steel which will be subsequently processed and supplied to RDSO.

A patent application has also been filed.

## 4. Estimation of residual creep life of thermal power plant components

The assessment of the remaining life of materials after elevated temperature service, received considerable attention over the recent years. The reason is well known; world-wide many power plants are achieving their design life time. From an economical point of view, it is necessary to postpone the replacement as long as possible provided that full safety can be guaranteed. An increasingly important aspect of the creep of engineering material relates to an assessment of the amount of damage which has taken place in components subject to high temperature service and an estimation of their remaining safe life. The different steps followed in the present approach for creep damage and life assessment of power plant components after elevated temperature service are :

1. Accelerated stress rupture tests of service-exposed materials for the purpose of evaluation of post-service properties;

2. Accelerated stress rupture tests of the unused material for the purpose of evaluation of pre-service (initial) properties. (In most of the cases, unused materials are not available and hence their pre-service properties cannot be evaluated experimentally. Under these circumstances, the property of the material having similar composition and thermo-mechanical treatment is to be taken from literature, international specification etc., if available, for the purpose of comparison.);

3. System data analysis of service exposed and unused material using different time-temperature parameters for creep life assessment of service exposed power plant materials;

4. Microstructural investigation on (*i*) unused material; (*ii*) service exposed material and (*iii*) creep rupture samples for creep damage assessment of service exposed power plant components and its correlation with remaining creep life.

The components received from various thermal plants for creep life assessment are :

(*i*) steam pipes from Neyveli Lignite Corporation;

(*ii*) platen superheater tubes from Badarpur Thermal Power Station;

(*iii*) steam pipes from Patratu Thermal Power Station;

(*iv*) carbon steel (ASTM 201) samples of an elevated temperature equipment from Engineers India Ltd.

## 5. Steels for short-term evaluation completed & submitted

(*i*) Strees rupture test on 10 samples, sponsored by Sivananda Steels Ltd., Ambattur, Madras.

(*ii*) Tensile test and stress rupture tests on the steel samples sponsored by Sivananda Steels Ltd., Madras.

(*iii*) Stress rupture tests on 9 steel samples, supplied by Sivananda Steels Ltd., Madras.

(*iv*) Hot tensile tests and stress repture tests on 6 steel samples supplied by Sivananda Steels Ltd., Madras.

(*v*) Creep tests on 2 steel samples of 21-4N steel to determine 1% creep strain, sponsored by M/s. Shama Engine Valves Ltd., Bhopal.

(*vi*) Hot tensile tests on steel samples, supplied by M/s. Indian Seamless Metal Tubes Ltd., Ahmednagar.

### 6. Novel thermocouple welding technique

A novel technique for thermocouple welding has been developed. The technique is simple, cheap and needs no welding torch or other complicated arrangements. It is based on the principle of electrolytic heating. The electrolyte required for the purpose can easily be prepared by mixing sodium carbonate with water in a glass container. Sodium chloride can also be used in place of sodium carbonate for making the electrolyte but due to its corrosive effect, in this case, care should be taken to wash the weld joint in fresh water thoroughly.

Anode is placed in the electrolyte and the thermocouple wires (twisted together) to be welded, which will act as cathode, is just dipped in the electrolyte and it is welded in a matter of seconds. The technique is suitable for welding practically all types of thermocouples or other suitable pair of metal wires. This principle of electrolytic heating can also be used for the purpose of heat treatment of metals and alloys.

## H. METALLURGICAL INVESTIGATION—FAILURE OF METALS & ALLOYS

| *Sl. No.* | *Nature of Investigation* | *Sponsored by* |
|---|---|---|
| 1. | Investigation on the failure of water pipe of High Rise Building | Electricity Board Complex, Madras |
| 2. | Failure analysis of Platen Superheater tube (CBIP/22) | Ennore Thermal Power Station |
| 3. | Failure of Superheater tube (CBIP/18) | -do- |
| 4. | Failure of Economiser tube (CBIP/21) | -do- |
| 5. | Metallurgical Examination of Boiler plates | Hiralal Jiwanlal Saw Mill, Indore |
| 6. | Failure of (*i*) Feed Bye-pass and (*ii*) Second-stage Superheater tube | Barauni Thermal Power Station |
| 7. | Failure of Telescopic Power of P & T at Randha, Seraikela | P & T, Calcutta |
| 8. | Failure of HK-40 Reformer Tube for Ethylene Plant Furnace | Engineers India Ltd., New Delhi |
| 9. | Investigation on the Metallurgical State of Health of Coke Chambers | IOC, Gauhati Oil Refinery |
| 10. | Failure of Compressor in Ammonia service | IOC, Barauni Oil Refinery |
| 11. | Failure of Platen Superheater tube (CBIP/27) | ETPS, Tamil Nadu |
| 12. | Failure of (*i*) Ball Mill Drive Shaft, and (*ii*) Rotor Bar of Boiler Feed Pump Motor | Patratu Thermal Power Station |
| 13. | Failure of Platen Superheater tube, Unit No. V (CBIP/25) | ETPS, Tamil Nadu |
| 14. | Metallurgical evaluation of imported Steel Plate contemplated for use in the second Hooghly Bridge Construction | Hooghly River Bridge Commissioner, Calcutta |
| 15. | Failure of Superheater tube (CBIP/29) | ETPS, Tamil Nadu |
| 16. | Failure of Convective Superheater tube (CBIP/30) | Korba Thermal Power Station |
| 17. | Investigation on the Failure of SF Rings used in Textile Machinery | Texmaco, Calcutta |
| 18. | Investigation on the Failure of superheater, economiser and water wall tubes of Unit No. 3 | Panki Thermal Power Station, Kanpur |

| Sl. No. | Nature of Investigation | Sponsored by |
|---|---|---|
| 19. | Failure of steam cooled water wall tube | Badarpur Thermal Power Station, NTPC |
| 20. | Failure of economiser tubes | IOC, Barauni Oil Refinery |
| 21. | Failure of economiser tubes of Harihar Polyfibers | Renusagar Power Co., Renukoot |
| 22. | In-situ metallographic examination of reactors | Hindustan Polymers, Visakhapatnam |
| 23. | Failure of Natural Gas Pipeline | IOC, Duliajan |

## I. MECHANICAL WORKING & TESTING

### 1. (*a*) Aluminium-mild steel-aluminium sandwich sheet

Hot roll bonding of Al-Ms-Al sandwich sheet has been developed. The parameters of diffusional heat treatment of the roll bonded sandwich sheet has been studied for satisfactory bond integrity.

Further work of hot-roll bonding of hot-dip aluminised MS sheet with Aluminium on both side is under progress. The effect of aluminised interlayer during diffusional annealing is under study.

Improved bond integrity has been observed due to the pressure of interlayer after diffusional annealing and heat treatment etc.

### (*b*) Fatigue testing

Fatigue testing of HLS-45 connecting Links; Sponsor—M/s. MAMC Ltd., Durgapur, West Bengal.

Fatigue testing of 50 connected links samples were conducted.

### 2. Development of contact material by melting technique

Studies on the composition containing 3.5 and 7.5 cadmium in silver have been made. Various parameters such as cadmium loss during melting, temperature for initiation of internal oxidation, effect of temp. on the micro-structure etc. have been standardised. Roll bonding parameters of Ag/AgCdO composites have also been standardised. The project is completed and the report is being prepared.

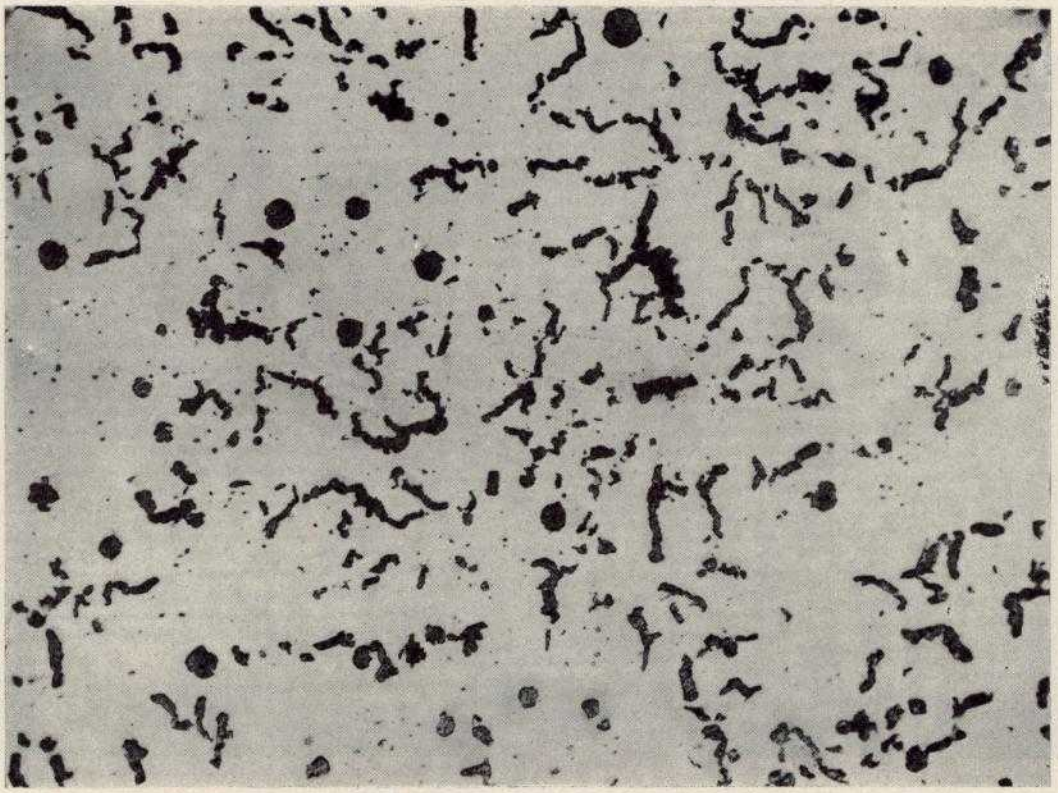
### Development of silver-base brazing alloy

The alloy selected for study contained Ag-60%, Sn-10%, Cu-30% with smaller additions of phosphorous. This is a corrosion resistant type of alloy due to the presence of phosphorous and finds wide application in sophisticated instruments. Processing parameters have been standardised. The project is completed and a report is being prepared.

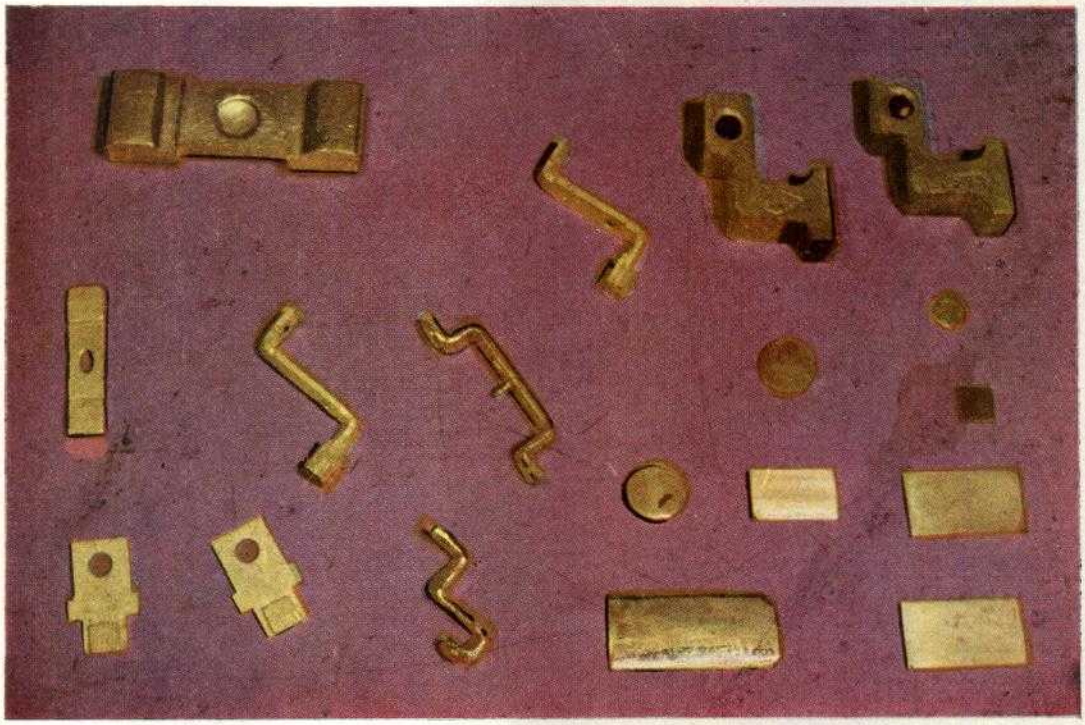
### Investigations on contact materials and silver brazing alloy

*1. Development of process know-how of silver copper/cadmium alloy*

Few heats of silver and copper containing 10 and 20% copper were made. Process parameters have been standardised. Physical and mechanical properties have been determined. The work on these two compositions is complete.



*Microstructure of compacted graphite cast iron (mild steel as base material)*



*Silver-cadmium oxide contact materials developed at NML for using in power sectors*

2. *Development of process know-how for preparation of silver brazing alloys*

The work on BAG1 and BAG2 containing silver-copper-zinc and cadmium was done. After melting and casting the slab, ingots were homogenised for different hours depending on the composition variation, surface dressed and then rolling schedule standardised for obtaining final products in thin sheet and foil.

3. *Service performance trials of contact materials and silver brazing alloys*

In order to evaluate the service performance trials, samples were prepared as per specification and composition given by the parties. These were supplied to M/s. Tisco, Bokaro Steel Plant and Bhilai Steel Plant.

Interim satisfactory report on their performance has been received.

4. *Development of W-Ag contact materials by P/M technique*

The formation of 75 W-25 Ag contact materials has been routed through conventional sinter infiltration technique, and the properties of the contacts so produced have been found as per specification.

Besides, the contact materials have also been produced by pressing both the powders simultaneously in the same die with a single thermal treatment in order to show greater advantages over the conventional technique.

5. *Development of nichrome alloys*

Work for the development of 60 Ni-16 Cr- and balance iron has been completed. The manufacturing process covering the melting, casting and mechanical processing of the alloy finally in the form of wire has been standradised. The physical and mechanical properties of the alloy including the life have been evaluated and found as per specification.

## J. FOUNDRY TECHNOLOGY

### 1. Development of compacted graphite cast-iron structure property correlation

(*a*) Extensive investigations were carried out to study the suitability of sponge iron and pig-iron as base charge for the production of compacted graphite iron using Fe-Si-Mg as an inoculant and Fe-Ti as denodulariser. Sponge iron from Orissa as well as from NML yielded CG iron and SG iron of improved quality. With Bhilai pig as base charge, CG iron also has been prepared successfully.

(*b*) Scheme of experiments for the production of CG iron in Fe-C-Al system has been done. A few heats also have been studied.

(*c*) The preparation of the base charge for production of CG in view of its properties to be achieved by heat treatment has been taken up.

### 2. Oils of vegetable origin as mould/core binders

Polymerisation characteristics of nine oils have been evaluated at different time-temperature combination by determining the tensile strength of sand specimens bonded with above oils. The experiment revealed that polymerisation of oils produce strengths which are comparable as obtained with linseed oil. However, with linseed oil, the bond strength is retained to wider range of temperatures which is a distinct advantage.

GLC studies with regard to unsaturation and UV IR studies with regard to structure have been carried out on 15 nos. of oils at RRL, Hyderabad.

### 3. Development of humidity resistance chemical additives for sodium silicate bonded sand system

Sodium silicate bonded mould/cores are generally regarded as not suitable for long-term

storage due to determination under high humidity. To reduce adverse effect due to high humidity, addition of some inorganic chemical compounds were made to study the strength properties of silicate-bonded sands at different relative humidity. The experimental results are encouraging.

## K. CORROSION STUDIES ON METALS & ALLOYS

### (*a*) Development of high output non-pollutant aluminium base sacrificial anode for cathodic protection

NML-HOPAL anode has been successfully developed with the view of substituting the imported anodes being used for the protection of off-shore oil platforms and the pipelines. The developed anode has potential of −1.2 V and life of 4.2 kg per Amp year with 96% current efficiency.

The technology has been transferred to 3 different parties.

### (*b*) Stress corrosion cracking of stainless steel in nitrate and chloride environment under sustained and dynamic tensile loading conditions

Initiation and growth of crack of AISI 304 steel was studied under static and dynamic tensile loading conditions. Both transgranular and intercrystalline cracks were observed. Special features on crack growth VS potential shift was noted to be a great deal of fluctuations in the Pot-Time during the test. This has been correlated to the formation and break-down of the film formation and intervention of IR in the system.

### (*c*) Studies on corrosion of Nickel and stainless steel in non-aqueous solvents

Corrosion behaviour of AISI 304 steel and pure nickel was investigated in 'protic', 'aprotic' and amphoprotic types of solvents. Effect of ions on corrosion behaviour of metals varies from solvent to solvent. Chloride ion is found to be more aggresive in protic than in other two types of solvents. Nitrate ions in general inhibit the corrosion whereas sulphate accelerates the attack.

## L. SURFACE COATING ON METALS

(*i*) The collaborative project between NML and PDIL, Sindri on "Development and performance evaluation of diffusion treated steels in fertilizer and chemical industries" was successfully completed and the joint terminal report was submitted.

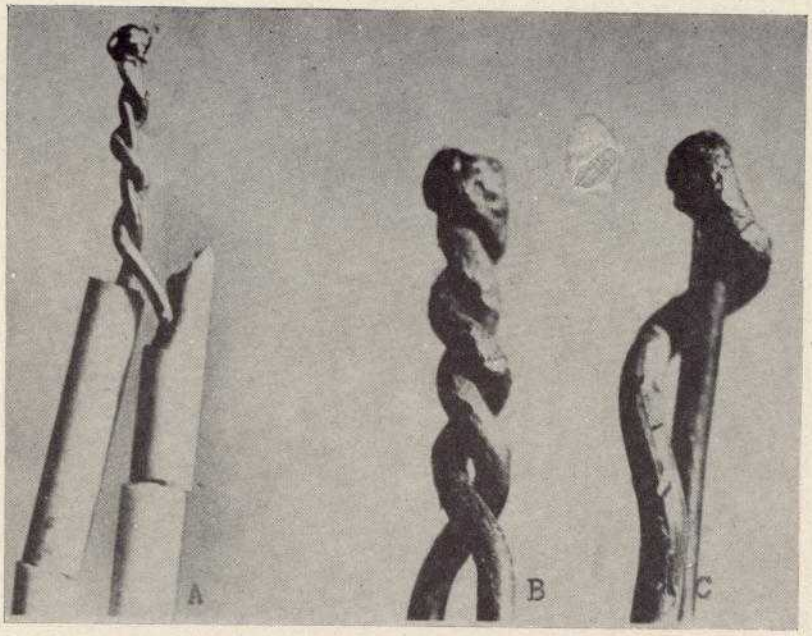
Aluminium Diffusion Treated Steel (ADTS) developed at NML was found to be on and par even superior to imported ALON heat exchanger tube material based on the laboratory hot corrosion tests in sulphurous atmosphere and also inplant exposure trials. The material was also tested by Indian Institute of Petroleum, Dehradun for its resistance towards Napthenic acid encountered in petroleum processing and it was found to be at least six times more resistant than uncalorised steel.

(*ii*) The project on "Metal Pigmented Primers for corrosion protection of steel" was completed on the experimental stage in March, 1986.

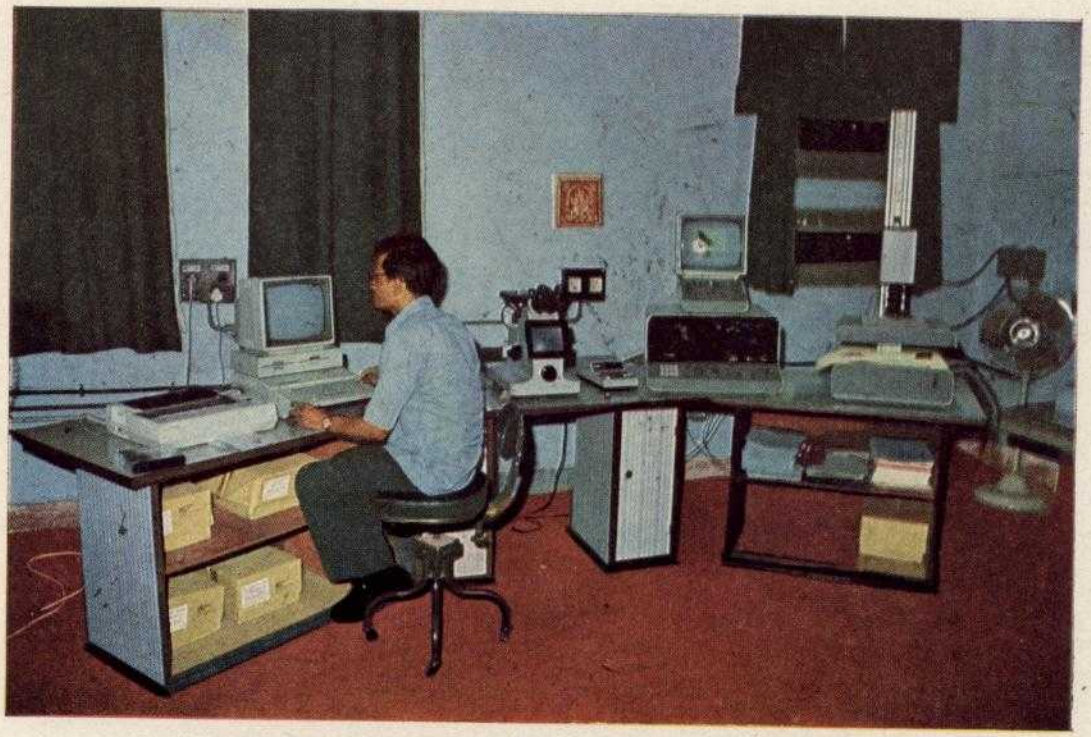
Prehydrolysed Ethyl silicate-40 having stability of a few months to serve as a binder for zinc rich primer could be prepared under controlled conditions of hydrolysis and the pot life of a mixed zinc paint using the said vehicle is about 6 to 8 hours. The primer cures in 2 to 3 hours and imparts excellent galvanic protection to steel. A number of compositions were prepared and evaluated for adhesion, galvanic protection and resistance to different organic solvents. The primers are composed of entirely inorganic materials besides zinc are not only good galvanic primers but also offers good resistance to organic solvents.

(*iii*) *Conserving energy in nickel deposition by AC potential*

The project was completed on laboratory scale. The newly developed process gives satisfactory deposits on steel, brass, copper, phosphor, bronze etc., at room temperature and lower concentration of the imported reducing agent (Sodium hypophosphite) compared to the conventional process in which the batch is heated to a temperature of 80-100°C. The various parameters for satisfactory deposits were optimised and the microhardness and microstructure of the deposits were determined.




*Thermocouple weld joints by electrolytic heating technique*



*Automatic image analyser for quantitative metallography*

(*iv*) *Exploratory projects*

(*a*) *Electrolytic nickel powder:* It was possible to produce electrolytic nickel powder with a spiky and dendritic structure.

(*b*) *Calorising of stainless steels:* Austenitic stainless steel grades AISI-304 and AISI-316 were calorized under optimum conditions using Fe-Al alloy powder and the oxidation resistance of calorised steels was studied at 1000°C and 1100°C for 20 hours. Further work is in progress.

(*c*) Development and evaluation of chromium boride paste for improvement of the wear resistance of steel.

The imported chromium boride paste obtained from SAIL (R & D) was chemically analysed and it was found to consist of mainly Cr and B with minor percentages of Silicon and Iron. The Cr:B ratio was found to be 1:1.15. X-ray analysis indicated the aggregate consists of CrB compound.

A few mild steel samples were coated with the said paste and fused with oxyacetylene/carbon arc to the surface and the microhardness of the coating was found to be 1350 VHN. Further work is in progress.

## M. ELECTRON MICROSCOPY

### 1. Structure of martensites in Cr-Mn-N austenitic steels

Crystallography of martensite in Cr-Mn-N austenitic steel have been studied by means of transmission electron microscopy. Defect analysis of various kinds of dislocation substructures have also been made.

### 2. Development of shape memory alloy

Few heats of shape memory alloy based on Cu-Zn-Al have been made and its crystallography were investigated by TEM. A new martensite structure 54R have been found with the help of direct lattice imaging and micro beam diffraction. This structure is thought to play an important role in minimising the irrecoverable strain. The development work is in progress.

### 3. Microstructural evaluation of creep resisting steels

$2\frac{1}{2}$Cr-1Mo, Cr-Mo-V, Creep resisting and Cr-Mo-V, Ti-B Bolting steel have been studied by TEM. The morphological difference in virgin and service exposed specimen have been assessed. In vanadium bearing steel, a new superlattice of vanadium carbide have been investigated. The partitioning of alloying elements in various kinds of precipitates have also been assessed.

### 4. Investigations undertaken for inhouse R&D programmes are as detailed below

(*a*) Calorised mild steel
(*b*) Polymetallic sea nodules
(*c*) Iron oxide powders
(*d*) Al-Li alloys
(*e*) Fe-Cr-Co permanent magnet alloys

## N. STANDARD REFERENCE MATERIAL & CHEMICAL ANALYSIS

Analysis was carried out on 2,533 samples for 7,049 radicals. A large number of samples of polymetallic nodules received from National Institute of Oceanography, Goa were analysed. Analytical services were rendered to many outside organisations both under public and private sectors as well as research institutions.

### Preparation of standard reference materials

These materials are prepared in Analytical Chemistry Division of the laboratory. Those samples are used in industries as well as research institutions for accurate and rapid analysis of raw materials and finished projects. They are also used for checking the developed analytical techniques.

Certification of Manganese ore and Ferro-Vanadium were completed and put to sale. Preparation of samples of low carbon steel and cast iron were taken up in addition to replenishment work.

## O. APPLIED BASIC PROJECTS

### 1. Use of the non-isothermal thermo-analytical model for correlating the actual operating parameters of a vertical retort unit for DRI production with the model incorporating geometrical, mechanical and dynamic similarity criteria

With a view to evaluating the mathematical model for correlating productivity as dependent operating parametric variable in vertical retort furnace. Experiments were conducted with (*i*) different iron ore/coal particle size; (*ii*) different iron ore/coal ratio, reacting under non-isothermal conditions akin to actual process. The work is in progress.

### 2. Development of shape memory alloys

Development of shape memory alloys based on Cu-Zn-Al system—estimate of their shape memory effect after thermomechanical treatment and possible introduction of two way memory has been initiated.

Forging and hot rolling conditions are being standardised.

### 3. Structure property correlation of calorised steels

The probing and bench scale experiments regarding the structure of calorised coatings have been performed using optical microscopy, microhardness profiles, X-ray and electron differaction work is in progress.

### 4. Study on the effect of chlorine and nitrogen bubbling on the solidification behaviour and properties of Al-Si and Al-Zn-Mg alloys

Diffuser model has been developed and a patent application is being made. The use of diffuser would enable to effect a non-polluting degassing process and also transfer the case structure.

### 5. Solidification behaviour of Al alloys and Al alloy composites

Composite production through the approach of casting from the semi solid state has not been done anywhere in India. Semi solid state casting enables retention of immiscible phases in a desired distribution pattern in the matrix, as has been experienced in earlier work in this laboratory. These materials showed improved resistance and thermal expansion characteristics as compared to casting from superheated melt. A few typical compositions based on Al-Cu-Mn and Al-Zn-Mg have been selected for studies in this set up. Lithium containing alloys also are to be studied for their solidification behaviour.

### 6. Structural/physical properties and phase transformation studies of supersaturated Al-Cu solid solution containing various amounts of Cr

The possibility of formation of Pseudo Crystalline structural aspects that are evolved in some rapidly frozen binary Al base alloys like Al-Mn are being studied.

### 7. High temperature oxidation studies of boiler super-heater tubing material $1\frac{1}{4}$ Cr—$\frac{1}{2}$ Mo and $2\frac{1}{4}$ Cr-1 Mo steels

The formation of different layers of oxides e.g. magnetite, hematite, wustite and other complex oxides were observed and found to be a function of working environment and condition in boilers.

# EXTENSION UNITS

## NML CENTRE, CSIR COMPLEX, MADRAS

The Centre completed successfully several important investigations such as, Beneficiation studies on Salem Magnesite (Phase I); Beneficiation studies on a Graphite Ore from M/s Dorr-Oliver, Madras; Magnetic separation studies on a Pegmatic Ore from M/s. Sakti Minerals Pvt. Limited; Briquetting studies on lime produced from a sample of Crystalline Limestone from M/s Industrial Chemicals & Polymers; Corrosion Studies on Boiler Tubes from M/s Seshasayee Paper & Boards Ltd., etc.

The NML Madras Centre has undertaken chemical analysis, Metallographic/Mineralogical Studies on various samples received from outside parties as also for the R&D programmes.

A brief resume of the various projects and activities is furnished below:

### 1. Beneficiation studies on Salem Magnesite (Phase I) sponsored by Mineral Development Board, New Delhi

Detailed beneficiation studies were conducted on different types of Magnesites occuring in the Salem area of Tamil Nadu. Five type samples were identified based on the nature of mineralisation and presence of Silica etc. The different type samples were collected from different mines in Salem. The type (B), which is being mined commonly was collected from two mines. Total six samples of Magnesite and also a sample from dumps were collected. The samples assayed from 16.18 to 31.87 per cent $SiO_2$. The Magnesite mineral content in the sample was varying widely from 9 to 85 per cent.

Primary crushing, washing and sorting studies were conducted on each type of sample up to 12 mm size. Heavy media separation studies were conducted on $-12$ mm$+3$ mm fractions of R.O.M. ore and middlings. Heavy Media Cyclone studies were conducted on $-3$ mm$+20$ mesh fractions. Flotation studies were conducted on $-20$ mm fraction after grinding to 60 percent $-200$ mesh. Based on the results of batch tests, pilot plant studies were conducted on all the Magnesite type samples.

Overall, it was found that not only the types presently mined, but also some of the ore types, which are not being mined, stack samples and the dump samples favourably responded to beneficiation methods. A generalised flow-sheet was drawn to treat all types of Salem Magnesites for the production of usable Magnesite concentrates and to increase the output to twice the present recovery.

### 2. Beneficiation studies on Salem Magnesite (Phase II) sponsored by Mineral Development Board, New Delhi

Based on the results of phase I, detailed project report preparation work was taken up and it is in progress.

### 3. Beneficiation studies on Graphite Ore sponsored by M/s. Dorr-Oliver, Madras

M/s Dorr-Oliver, Madras requested NML Madras Centre to find out the suitability of Dorr-Floatation Cell and Dorr-Rake Classifier for beneficiation of Graphite ores from Sivaganga, Tamil Nadu. The classification tests were carried out on a Rake Classifier 1′ width and 16′ length and floatation tests were carried out in LA500 Dorr type cell. It was found that the Rake Classifier and Dorr-Floatation Cell were also giving satisfactory results. These tests were done at the instance of M/s. TAMIN.

### 4. Magnetic separation studies on Pegmatic Ore sponsored by M/s. Sakti Minerals Pvt. Limited

About 20 Kg of Pegmatic Ore was sent by M/s. Sakti Minerals Limited to crush, grind and magnetically separate the contaminating minerals. The ground non-magnetic ore was screened over 100 and 150 mesh and 5 gm portion of each of the screened sample was heated to

1000° to 1100°C and it was observed that heating above 1000°C led to the formation of desired colour. The results were reported.

### 5. Briquetting studies on Lime produced from a sample of crystalline limestone, sponsored by M/s. Industrial Chemicals & Monomers Limited, Tamil Nadu

The object of this investigation was to develop a method to produce briquettes of quality and strength suitable for Calcium Carbide production. The samples essentially consisted of crystalline calcite varying in size from 25 mm to fines and analysed 55.27 per cent CaO and 1.25 per cent $R_2O_3$. Optimum calcination conditions in a batch muffle furnace to obtain soft burned, for production of briquettes of reasonably good strength was 1.5 ton/sq. cm. Use of additives, such as $Ca(OH)_2$, Lignite, Wax, Kerosene and Sugar in improving the life of briquettes were studied. The briquettes usind 10 percent lignite withstood 7 hours without crumbling.

### 6. Column-Electrofloatation

Earlier experiments on the beneficiation of Chalcopyrite fines by Electrofloatation were found to be quite encouraging with almost 85-90 per cent recovery in a single stage operation. As a partial modification of this set up to improve the grade, Column-Electroflotation Cell (height 75 cm. dia 7.0 cm.) was developed using nickel coated SS wire mesh as an anode.

Trial experiments conducted on the fines of Chalcopyrite from Chitradurga using Potassium Ethyl Xanthate as a collector resulted in a better grade. The grade of the concentrate was fonud to improve up to 14 percent of Cu, compared to 8 per cent of Cu obtained by electroflotation alone. Studies on the optimization of parameters like current density, effect of auxiliary air conditioning time, collector concentration etc., are under progress.

### 7. Beneficiation studies on Malanjkhand Copper Sulphide-Oxide Mixed Ore

Initial experiments conducted on the beneficiation of above sample revealed the need of some special collectors. Experiments using Diethyl dithiocarbomate (DTC) complexing agent as a collector was found to be effective. Work is under progress.

### 8. Beneficiation of Scheelite Ore from Jaurasi, Uttar Pradesh

Beneficiation tests on the low-grade Scheelite from Jaurasi were attempted. From the initial tests, it was found that the mineral was finely dessiminated and content of $WO_3$ is as low as 0.1 per cent to 0.08 per cent. Overall response by physical separation methods was found to be not encouraging. Various flotation methods like, Hot flotation (at different temperatures), Electrofloatation (at various current densities) and Electrofloatation with auxiliary air supply were adopted using Oleic Acid, Naphthenic Acid-Oleic Acid 1 : 1 ratio (Mixed Collector System). Improved results (recovery 40 per-cent Grade 3 per-cent $WO_3$) were obtained by using mixed collector system in combination of Electrofloatation.

### 9. Tests on grinding and adsorption of activated carbon samples were conducted for M/s. Tamil Nadu Alloy Industries, Madras

Investigations on (*i*) Failure of boiler tube sponsored by M/s. Seshasayee Paper & Boards Ltd., (*ii*) Failure of water wall boiler tubes sponsored by Tuticorin Thermal Power Station and (*iii*) Failure of liners in Generator sets sponsored by Indian Organic Chemicals Ltd., Madras were carried out.

### 10. Heat treatment, metallographic studies, mineralogical studies, calibration of thermocouples, refractory testing etc.

The centre conducted the above assignments on behalf of various industrial units and organisations. Mineralogical studies were conducted on samples of ores and minerals received for beneficiation investigations. 66 ferrous and non-ferrous alloys were studied to evaluate their microstructure. 11 samples were tested for hardness and heat treatment was conducted on 6 samples. 4 cases of failure of metal components during service or processing were investigated. 78 Cr/Al thermocouples were tested for their accuracy and test certificates furnished to the concerned parties. 780 samples for 2371 radicals were analysed for various industries and Unit's R&D Programmes. Microhardness tests were conducted on behalf of two firms. Refractory tests were conducted on behalf of two firms.

# FIELD STATIONS

## NML FIELD STATIONS AT HOWRAH, BATALA AND AHMEDABAD

During the period under review, the Field Stations had rendered the necessary technical services to the engineering and foundry industries particularly the small scale units in and around their respective locations. These services comprised of chemical analysis (both quantitative and qualitative) of various metals and minerals, mechanical testing of metals and alloys, testing of foundry moulding sands and bonding clays etc., on the spot study of the foundry production problems and their remedial measures, improvement in quality according to stringent specifications and export purposes etc.

A comparative statement of the work done by the Field Stations at Howrah, Batala and Ahmedabad during 1985-86 has been furnished in the table below:

| Sl. No. | Particulars | 1985-86 | | |
|---|---|---|---|---|
| | | Howrah | Batala | Ahmedabad |
| 1. | Chemical Analysis: | | | |
| | (*a*) No. of samples | 776 | 295 | 903 |
| | (*b*) No. of radicals | 3,414 | 785 | 3,349 |
| 2. | Mechanical Testing: | | | |
| | (*a*) No. of samples | 869 | 127 | 4 |
| | (*b*) No. of tests | 869 | 127 | 4 |
| 3. | No. of Technical enquiries | 9 | 179 | 96 |
| 4. | No. of Foundry visits | 5 | 130 | 2 |
| 5. | Total no. of units served | 320 | 91 | 402 |

In addition to the above, the Howrah Field Station had conducted metallography tests on fifteen samples while the Batala Field Station had conducted tests on one hundred and twenty-three samples. The metallography tests at the Howrah Field Station comprised mostly on the distribution of graphite and matrix structures of cast iron, malleable iron SG iron etc., and evaluation of microstructure of grinding media ball samples.

The Howrah Field Station had conducted investigation on grinding media ball samples for evaluation to examine its conformity to IS: 6079-1980 specification. A report on the findings of the wide variations in respect of chemical composition, hardness values and microstructure with that of the standard specifications, along with the comments was sent to M/s. Fertiliser Corporation of India Limited, Talcher Unit, Orissa. The Howrah Field Station had conducted a number of tests to find out the feasibility of using the sodium silicate obtained from rice husk at the CGCRI, Calcutta for foundry purpose.

# ENGINEERING SERVICES

## ELECTRONIC ENGINEERING

### A. Instrumentation of Projects

(*i*) *Corrosion*

Corrosion meter designed earlier has been redesigned as some of the components were not available in India, some components including MOS-FET operation amplifier (integrated circuit) have already been procured.

(*ii*) *Analytical*

Atomic Absorption Spectrophotometer SP & 100 (with microprocessor control) has been installed made by M/s. Philips.

### B. Major maintenance jobs done

(*i*) XRF (*ii*) XRD (*iii*) AAS (*iv*) DTA (*v*) Potentiostat (*vi*) Vibrophore.

## ELECTRICAL ENGINEERING

### 1. Design and development of electro heat and electric control system

(*i*) Work on development of arc plasma reactor for use in metallurgical processes was continued. With a view to developing arc plasma torches, design parameters were collected.

(*ii*) Design and fabrication of five numbers 3-Zone isothermal electric resistance furnaces for multispecimen Creep Testing machines were carried out.

(*iii*) Design and fabrication of muffle, tube and pot furnaces up to the temperature range 1150°C and power rating of 20-15 KW were taken up for various metallurgical research and development work.

### 2. Engineering systems design, planning, installation and commissioning

Design of power distribution and air-conditioning systems, preparation of detailed specifications and layouts, planning, installation and commissioning were carried out with respect to various jobs such as:

(*i*) Installation of power supply system and control units of new equipment in the laboratory.

(*ii*) Replacement of unserviceable power supply system.

(*iii*) Installation of 200 TR water cooling tower, FRP construction and induced draft type for air-conditioning system of Central Creep Testing Laboratory.

### 3. Establishment of facilities for R & D work

Preparation of detailed specification and scrutiny of quotations with regard to high temperature furnaces in the range of 1400°-2000°C were carried out.

### 4. Preventive maintenance and breakdown repairs

Preventive maintenance and breakdown repairs were carried out through proper planning, regular inspection, execution and monitoring of various electrical equipment while in service, fault shooting and repairing in respect of electrical and air-conditioning equipment of the Laboratory, its Pilot Plants and residential areas, comprising of high tension sub-stations, resistance

furnaces, arc furnaces, rectifiers, electric motors and their control centres, compressors, water pumps etc.

### 5. Forecasting and procurement of spare parts

Forecasting and procurement of spare parts for power distribution system, temperature and humidity control system, melting facilities, metal testing facilities, Pilot Plants etc., were carried out.

## CIVIL ENGINEERING

The civil engineering jobs pertaining to modification of the old auditorium and lounge, for extending the library accommodation facilities, construction of water supply line to the MS Flats in the Agrico Area, usual maintenance jobs like white washing and painting of the double storey building FGH types, Tuiladungri, MCRS buildings at Digha, old 'D' type flats at Agrico area, 'B' type Bungalows at the pipeline area and the NML main laboratory building work completed.

Water proofing treatment to NML main building, Ferro-alloy Plant and Stores Shed etc., State Bank of India, NML branch building were executed.

Various other jobs like renovation of Community Development Hall at Agrico and Tuiladungri area, providing cattle guard at Agrico, repairing/replacement of doors and windows, rewinding of motors for water pumps, opening the doors in the MMT and providing Marblex PVC tiles at the Library room at NML were completed during the period.

# RESEARCH PLANNING

## ANNUAL PLAN

The Annual Plan Document 1986-87 including RE 1985-86, BE 1986-87 and the Seventh Five Year Plan projection 1985-86 to 1989-90 was prepared based on the R&D programme finalised by the RAC/EC of the laboratory.

## RESEARCH ADVISORY COUNCIL

During the period two RAC meetings and 6 RAC panel meetings were held for reviewing the R&D programme. Follow-up actions are being taken based on their recommendations.

## R&D DOCUMENTS

The following documents were prepared for projecting the activities of CSIR laboratories as desired by CSIR:

1. R&D activities during 1980-85.
2. Development of manpower.
3. Know-how developed and licensed.
4. Quantification of impact of work done.
5. Cash flow.
6. Services rendered to Government and other agencies 1980-85.
7. Thrust R&D Projects.
8. R&D projects/Sponsored projects operated during the Sixth Plan period.
9. Performance review with targets/milestones.

# RURAL DEVELOPMENT

NML Scientists actively participated in the Science and Technology Conference and exhibition organised by Marathwada Tantranya Parishad at Latur, Maharashtra State during 8th to 10th December, 1985. The Conference was inaugurated by Honourable Shri Shivraj Patil, Union Minister of State for Science & Technology & Vice President, CSIR. Exhibits on technologies as applicable to rural sector such as dental amalgam alloy, copper powder, electro-plating, aluminium alloy conductors, technology for reduction of losses encountered in aluminium utensil manufacture etc., were displayed and details were presented by lectures to the delegates.

A national Conference on Applications of Science and Technology for Rural Development in Gujarat State and an exhibition were organised by Govt. of Gujarat, CSIR, CART and DST on 31st December, 1985 and 1st January, 1986 at Porbandar. The objective was to know the present status of S&T applications in rural development in Gujarat with special reference to three identified areas and new technology for selected areas. The conference was inaugurated by Shri Shivraj Patil, Honourable Minister of State for S&T and Vice President of CSIR and presided over by Shri Amarsingh Chaudhary—Honourable Chief Minister, Gujarat State, Shri Mahant Vijayadasji, Hon'ble Minister for Agriculture & Rural Development and Shri Hasmukh Patel, Honourable Minister of State for Education, Gujarat State addressed the conference. NML exhibited technologies on mini cupola, improved chullah for metallurgical purposes, utilisation of bentonites, moulding sands, fishing hooks etc. The expertise and technologies available for rural areas was highlighted in a lecture by the NML Scientist to the delegates representing different categories like professors, scientists, industrialists, field workers, researchers, economists, engineers, architects, bank managers, educationists, Central and State Government officials etc.

NML also participated in the 'Shivir' (workshop) organised by Vikash Bharati, Bishnupur, Dist. Gumla, Bihar on 29th and 30th May, 1985. As requested by Vikash Bharati, NML had prepared a proposal costing Rs. 4 lakhs to be submitted by Vikash Bharati to the sanctioning authorities for approval for setting up a common facility centre for research, demonstration, training and popularisation of improved and conventional agricultural implements. The proposal has since been approved by DST & CART as intimated to NML by Vikash Bharati.

NML actively participated in the Workshop-cum-Exhibition on 'Science and Technology for Rural MP' held at Bhopal, on 22nd and 23rd February, 1986. NML expertise and technologies including those applicable to rural sector were exposed in the seminar and exhibition, which was well received.

## PATENTS

The following patents were filed:

| | *Title* | *Inventors* |
|---|---|---|
| 1. | Improvements in or relating to production of pure magnesium carbonate from magnesite. | Prof. V. A. Altekar<br>Gurdial Singh<br>M. L. Dey<br>Dr. N. Dhananjayan |
| 2. | Process for the preparation of a stabiliser to inhibit autocatalytic decomposition of hydrogen peroxide in sulphuric acid-peroxide mixture containing metal ions produced during bright dipping and etching of copper and copper based materials. | Dr. D. D. N. Singh |
| 3. | A process for production of electrolytic and activated manganese dioxide. | Dr. N. Dhananjayan<br>P. L. Sengupta<br>S. C. Aush<br>P. K. Sinha |
| 4. | Improvements in or relating to process for preparation of an inhibitor suitable for pickling of steel structures in hydrochloric acid at room temperature. | Dr. Inder Singh<br>Dr. V. A. Altekar |
| 5. | A process for the preparation of catalysed oxygen scavengers suitable for prevention of metallic corrosion in systems using different grades of waters. | Dr. Inder Singh<br>Dr. V. A. Altekar |
| 6. | Improvements in or relating to coat low carbon steels and low alloy steels with aluminium diffuse zones. | Dr. P. Prabhakaram<br>A. N. Mukherjee<br>T. L. Sharma |
| 7. | An improved process for degassing of aluminium and its alloys with the objective to minimising pollution. | C. S. Sivaramkrishnan<br>R. K. Mohanti<br>Dr. Rajendra Kumar |

# TECHNICAL LECTURES

The following lectures were held:

| | *Speaker* | *Subject* |
|---|---|---|
| 1. | Chem. Ing. Tzvetan Marinov Dorbev<br>Research Assistant<br>Institute of Physical Chemistry<br>Bulgarian Academy of Science<br>Sofia, Bulgaria | Multi-layer Copper-nickel Chromium coatings |
| 2. | Dr. (Mrs.) Erzsebet Kovacs Csetenyi Head of Laboratory for Physical Measurements<br>Hangalu Engg. and Development Centre<br>Budapset, Hungary | Phase transformation in Al-Zn-Mg alloys |
| 3. | Dr. DENES BULKAI<br>Research Fellow<br>Hangalu Engg. and Dev. Centre<br>Budapset, Hungary | Latest development on extraction of alumina by Bayers process |
| 4. | Dr. Shyam Dighe<br>Project Manager<br>Westinghouse Electric Corpn.<br>Pttsburgh, USA | (*i*) Materials for ocean thermal energy conservation<br>(*ii*) Phosphoric Acid fuel cells and plasma arc in foundry technology |
| 5. | Dr. Kamaleswar Upadhaya<br>Prof. University of Illinois<br>Chicago, USA | Kinetics and Mechanism(s) of reduction of iron oxide from liquid slag by carbon saturated iron |
| 6. | Dr. Madhukar Nilmani<br>Lecturer in Extractive Met.<br>Dept. of Chem. Engg.<br>University of Melborne<br>Australia | Some industrial investigations in copper converter |
| 7. | Prof. Nikhil Sarkar<br>Head, School of Dentistry<br>Lousiana State University, USA | Dental metals and materials |
| 8. | Mr. Daniel Gnanamuthu<br>Member of Technical Staff<br>Rockwell International Science Centre<br>California, USA | Processing of materials with $CO_2$ Laser Beams |
| 9. | Prof. T. V. Pillai<br>University of Pennsylvania, USA | Current issues on Science and Technology |

## DISSEMINATION OF INFORMATION

NML continued to interact with industries and forwarded the non-technical note and other literature relating to various technologies and expertise of NML to interested industrialists, Government Organisation and Entrepreneurs etc.

A video film on 'Metals in National Development' was prepared and a Russian version was sent to the Moscow Exhibition through CSIR.

## Computer Facility

A number of interesting programmes like statistics, solution of linear/non-linear equation, multiple regression etc., have been suggested by the scientists, Programmes for the technical data of the investigations, personal file, salary bill of the staff etc. have also been studied.

## NML Technical Journal

The research/investigations carried out in various fields are brought out regularly in the House Journal.

## Documented Survey on Metallurgical Development

Monthly abstracting service of the laboratory on leading metallurgical articles drawn from over 600 journals received in the library have been made as usual.

## Annual Report

Annual Report for 1983-84 was prepared and published. The Annual Report for 1984-85 has been prepared and sent to the press.

## NML Newsletter

The issues of NML Newsletter were brought out and published.

## Newspaper clipping service

Daily newspaper including commercial and business papers were scanned and items of industrial and R&D importance, Governmental industrial policies, scientific and industrial inventions etc., were classified and departmentally circulated.

## Library Services

*1. General*

Out of a total grant of Rs. 6.28 lakhs for the year, the Library has procured 350 books and subscribed nearly 250 periodicals on metallurgy and allied subjects. In addition to this nearly 150 publications and 180 periodicals were received on exchange/complimentary basis. A special collection of 121 books worth 2400 dollars under Overseas Development Administration (ODA) Book Presentation programme of British Council Library has been completed.

*2. Resource Sharing Services*

The total collection of documents as on March 1986 was 50,000 approx. This figure includes only books/reports/specifications and bound volumes of journal.

The document collection was used by 300 staff members of NML and also by the staff members of 6 other organisations who have been admitted as 'Institutional Members' of the Library. In addition a large number of organisations and individuals throughout the country shared the resources of NML Library through the supply of xerox copies.

*3. Expansion Programme*

The shifting and re-arrangements work in the new space made available to the Library has been completed during the year.

To facilitate resource sharing programme, the Library has finalised the formalities for purchasing one plain paper copier for exclusive use in the Library.

Under the expansion programme of Library, processing for procurement of one microfilm fische reader printer is in progress.

*4. Development of Man-power Resources*

Sr. Library Assistant attended the training course for two weeks on 'Abstracting and Indexing' organised and held at NISIET, Hyderabad.

**Visit of Foreign Scientists under Science & Technology Programme**

1. Chem. Ing. Tzvetan
   Marinov Dobrev
   Institute of Physical Chemistry
   Bulgarian Academy of Science
   Sofia, Bulgaria

2. Dr. (Mrs.) Erzsebet Kovacs Csetenyi
   Head of Laboratory for Physical Measurements
   Hangalu Engineering and Development Centre
   Budapest, Hungary

3. Dr. Denes Csaba Bulkai
   Research Fellow
   Hangalu Engineering & Development Centre
   Budapest, Hungary

4. Dr. Shyam Dighe
   Project Manager
   Westinghouse Electric Corporation
   Pittsburgh, USA

5. Dr. Kamaleswar Upadhaya
   Prof., University of Illinois
   Chicago, USA

6. Dr. Madhukar Nilmani
   Lecturer in Extractive Metallurgy
   Dept. of Chemical Engineering
   University of Melbourne, Australia

7. Prof. Nikhil Sarkar
   Head, School of Dentistry
   Lousiana State University, USA

8. Mr. Deniel Gnanamuthu
   Member of the Technical Staff
   Rockwell International Science Centre
   California, USA

9. Prof. T. V. Pillai
   University of Pennsylvania
   USA

10. Dr. Eui-Tin Jun and
    Dr. Sekyung Lee — *Delegates from Republic of Korea*

# DEPUTATION AND TRAINING

1. Shri H. Patnaik went to West Germany on deputation under CSIR-DAAD Exchange of Scientists programme for a period of 2 months to study the parameters for Carbonising Sponge Iron in Rotary Kiln. He also visited a number of technical institutes.

2. Mr. R. Singh, Scientist visited West Germany under CSIR-DAAD Exchange Programme, February-April, 1985.

# CHAIRMANSHIP/MEMBERSHIP OF EXTERNAL BODIES/ASSOCIATIONS

| | | |
|---|---|---|
| 1. | Dr. M. R. K. Rao, Scientist | Chairman, Indian Ceramic Society, Jamshedpur Chapter; Fellow, Indian Institute of Ceramics, Calcutta; Member, Panel on Refractories, DGTD; Member, GSI Refractories Committee. |
| 2. | Mr. P. C. Sen, Scientist | Secretary, Indian Ceramic Society, Jamshedpur Chapter. |
| 3. | Dr. K. K. Singh, Scientist | Member, Institute of Ceramics, UK. |
| 4. | Dr. N. Dhananjayan, Scientist | Research Member, International Battery Material Association (IBA), Cleveland, Ohio, USA. |
| 5. | Mr. B. K. Saxena, Scientist | Hony. Secretary, Indian Institute of Metals, Jamshedpur Chapter; Hony. Secretary, Institution of Engineers, Jamshedpur Local Chapter. |
| 6. | Dr. L. P. Pandey, Scientist | Fellow of Institution of Chemists (India); Chairman of an analytical committee, ISI:SMDC:35. |

# APPENDIX I

**Papers Communicated, Presented and Published**

1. Experimental processing of some Indian clays for commercial purposes—By M. V. Ranganathan and N. Chakravorty.

2. Flow properties of Granular Solids—By S. Sivaiah, S. Rafiuddin and N. Chakravorty.

3. Sinter making Russian Practice—By S. C. Maulik. Papers 1 to 3 were prepared and sent for publication in the NML Technical Journal.

4. Mineral characterisation and processing of some Indian high phosphorus manganese ores at NML—By P. D. Prasada Rao, A Peravadhanulu and N. Chakravorty for presentation in the National Workshop on removal of phosphorus from manganese ores to be held at RRL, Bhubaneswar shortly.

5. Characterisation and beneficiation of Indian Manganese Ores studies at NML—By P. D. Prasada Rao, B. Banerjee and A. Peravadhanulu, for inclusion in a monograph by P. G. Centre, ore mineral processing, Nandahalli, Karnataka.

6. A note on 'Thermal analysis studies on ores and minerals at NML'—By A. Peravadhanulu for Indian Thermal Analysis Society, BARC, Bombay.

7. Approach to an appropriate route for beneficiation of low grade phosphate occuring in tribal areas—By T. C. De and N. Chakraborty.

8. Graphite mineral development in the tribal areas of Andhra Pradesh—A review on the present status—By S. Sivaiah, A. Peravadhanulu and N. Chakravorty. Papers 7 & 8 for presentation at the Seminar to be held in Bhopal on 'Mineral Development in Tribal Areas'.

9. Specific surface determination of fine particles—A comparative study on two different methods—By S. Sivaiah, J. P. Srivastava, D. M. Chakrabarti and N. Chakravorty.

10. Some considerations on design of a simple sub-sieve sizer—By S. Rafiuddin.

11. Flocculation as a tool for beneficiation of mineral fines—By P. N. Pathak et al.

12. Use of ultrafines in iron ore sintering—By S. C. Maulik and N. Chakravorty. Papers 9 to 12 prepared for presentation at the National Seminar on 'Fine particles processing, to be held at the Indian School of Mines, Dhanbad on 11th and 12th August, 1986.

13. Improvement in the manufacturing techniques of tar bonded basic refractories—By K. C. Ray, B. Chatterjee, P. C. Sen & M. R. K. Rao submitted to the task force committee of the SAIL-CSIR Project.

14. On the structural characteristics of copper based shape memory alloy—S. R. Singh and O. N. Mohanty. Presented at the 39th Annual Technical Meeting of IIM, Jamshedpur, 1985.

15. Effect of binders on the strength of partially calcined magnesia compacts—By K. C. Ray, B. K. Mittra, P. C. Sen & M. R. K. Rao. The paper was presented in the 50th Annual Session of Indian Ceramic Society held at Calcutta during 7-9th February, 1986.

16. Silica refractories—By P. C. Sen.

17. Magnesite and Chrome-magnesite refractories—By P. C. Sen.

18. Special refractories—By K. K. Singh.

19. Dolomite refractories—By P. C. Sen. Papers 16 to 19 presented in the Appreciation Course on Refractories Technology during 9-12th December, 1985 at NML.

20. Emerging trends in steel plant refractories—By K. K. Singh and M. R. K. Rao. Presented in October, 1985.

21. Recovery of copper, nickel and cobalt from ammonical leach liquor obtained by direct leaching of sea nodules—V. Kumar, R. K. Jena, B. D. Pandey, D. Jha, A. K. Nayak, D. Bagchi and D. D. Akerkar. It has been communicated for publication in Trans. of IIM.

22. Development of process for recovery of cobalt from ammonical raffinate solution obtained by solvent extraction of the solution produced in the leaching of polymetallic manganese sea nodules—A. K. Saha, Z. H. Khan and D. D. Akerkar. Papers 21 & 22 presented at IIM Technical Meeting held at Jamshedpur on 14-17th November, 1985. It has also been communicated for publication in Trans. of IIM.

23. Separation and recovery of copper and zinc by solvent extraction, electrowinning from the sulphate leach liquor of complex sulphide ore—R. K. Jana, B. D. Pandey, V. Kumar, M. G. Bodas and D. D. Akerkar. Paper presented at 3rd National Symposium on 'Separation Techniques' at Waltair on 9-11th January, 1986 and will be published in proceedings.

24. Energy conservation in ferro-alloy industry—Premchand and D. D. Akerkar. Published in the proceedings of the energy conservation in process industries held on July, 1985 at Roorkee.

25. Development of reduction roast ammonia leaching process for recovery of nickel and cobalt from lateritic nickel ores in India including nickel bearing overburden of chromite mines—D. D. Akerkar, M. S. Mahanty, A. K. Saha and V. A. Altekar. The International Seminar on Laterite, at Tokyo, Japan, 14-17th October, 1985. It has been published in the proceedings.

26. Instrumentation and control of reducing type electric furnaces for production of ferro-alloys—D. D. Akerkar, P. K. Bagchi and A. P. Choudhury. Presented at the Symposium on Analytical Instrumentation and Techniques in electro-chemistry, held from December 5-7, 1985 in CECRI, Karaikudi.

27. Some observations on the processing of Polymetallic sea nodules at NML—D. D. Akerkar. Presented and published in the proceedings on International Conference on Aluminium—85 at New Delhi.

28. Development of solvent extraction process for the production of non-ferrous metals at NML—D. D. Akerkar. Presented at the National Symposium on Solvent extraction held on 19-20th November, 1985 at A. C. College of Technology, Madras.

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33. Appraisal of the Emerging Technologies for pig iron production under Indian conditions. A. M. Pande, K. N. Gupta.

34. Kinetics and mechanism of reoxidation behaviour of solid reductant based DRI—N. S. Sahota, K. N. Gupta.

35. Characteristics of burden and their importance to blast furnace performance and operation—By K. N. Gupta. Papers 33 to 35 presented at the Annual Technical Meeting of IIM, 14-17th November, 1985.

36. Analysis of non-isothermal kinetic data generated under arbitrary temperature fluctuation—S. Prakash, K. N. Gupta, H. S. Ray. Proceedings of the Fifth National Symposium on Thermal Analysis held on 20-22nd December, 1985 at IIT, Kharagpur.

37. Use of $\alpha$-t plots in comparative estimation of emerging consumption and production rate—S. Prakash, K. N. Gupta, H. S. Ray. Int. Conference on Progress in Metallurgical Research, Fundamental and Applied Aspects, held on 11-15th February, 1985.

38. A bench scale experiment to simulate the vertical retort process for production of DRI—S. Prakash, K. N. Gupta, H. S. Ray. Iron making steel making, Res. Vol. 2186.

39. A moving bed experiment for simulation of iron ore reduction by coal in a continuous vertical reactor—S. Prakash, K. N. Gupta. Presented in Int. Conference on 'Progress in Met. Research Fundamental and Applied Aspects, held between February 11-15, 1985 at IIT, Kanpur.

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41. Reduction of iron ore under rising temperature and fluctuating temperature conditions—S. Prakash et al (communicated for publication in Thermochim Acta).

42. Iron ore sinter as iron burden in Indian Blast Furnaces—A. K. S. Mahapatra, M. C. Goswami, K. N. Gupta. Presented at the Seminar on Sintering of Iron ores held at Bokaro Steel City on 8th May, 1986.

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54. Low Cobalt Fe-Cr-Co permanent magnets by deformation aging technique.

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57. Metallurgy in thermal power plants engineering and operation—R. Singh and R. Kumar, Metals, 1985.

58. Some observation on anticipation of the remaining life of service exposed steam pipe in thermal power plants—R. N. Ghosh, R. Singh and R. Kumar—to be presented at the forthcoming International Conference on Creep: April, 1986, Japan.

59. Some observations on the use of time-temperature parameter for predicting remaining life of service-exposed steam pipeline of thermal power plants—R. N. Ghosh, S. Choudhury, R. Singh and R. Kumar.

60. Studies of thermal durabilities of indigenous foundry clays—R. R. Dash, S. Ghosh, S. K. Sinhababu, G. N. Rao.

61. Sand testing—a tool to preduct moulding behaviour—S. Ghosh, S. K. Sinha, T. A. Beck, G. N. Rao.

62. Production and properties of CG and SG iron from sponge iron—R. R. Dash, O. N. Mohanty Papers 59 to 62 presented at NMD 1985 at Jamshedpur.

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65. Studies on characterisation of different samples of poly-metallic sea nodules by electron optical instruments—S. Banerjee, S. R. Singh, S. K. Bose, A. K. Nayak, D. D. Akerkar. Presented at the National Seminar on Processing of Manganese nodules, R. R. L. Bhubaneswar (March, 1985).

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79. Pyrrole-2-Thiocarboximide as a new gravimetric reagent for nickel—L. P. Pandey and A. C. Basak, Jour. Inst. Chemists (India), July, 1985, p. 141.

80. Complexometric determination of manganese in manganese ores using thomalic acid indicator—V. N. Choudhury, N. N. Ganguly and L. P. Pandey, J. Inst. Chemists (India), 1985, 58, 216.

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83. Instrumentation and Control in the Hydrometallurgy of polymetallic sea nodules—D. D. Akerkar, A. P. Bhattacharya and P. K. Bagchi—Symposium on Instrumentation and Process Control for optimisation in metallurgical processes, IIM, Nagpur Chapter, 15-16 February, 1986.

84. Studies on syntactically coalesced silicon carbide polytype structures—Crystal Research and Technology, 19, (1984) 527, S. R. Singh.

85. Lattice imaging studies on structure and disorder in SiC polytype. Presented at the Instl. Conf. Crystal growth and Characterisation of polytype structures—Marseilles, France (July, 1984), S. S. Rai, S. R. Singh and G. Singh.

# APPENDIX II

## RESEARCH, INVESTIGATION & SURVEY COMPLETED AND REPORTS PREPARED

### Research Reports

1. High strength medium conductivity Aluminium Alloy Conductor NML-PM-215—R. Kumar, K. Lal, A. K. Bhattamishra (R/R 431/85).

2. Studies on sea-water magnesia produced by CSMCRI, Bhavnagar, Gujarat—N. N. Mathur, K. C. Roy, A. Dasgupta, P. C. Sen and M. R. K. Rao (R/R 432/85).

3. Studies on self setting sand process developed at NML through orthogonal array design of experiements—R. R. Dash, T. A. Beck, H. Patnaik (R/R 433/85).

4. Statistical and structural analysis of Indian Foundry Clays—R. R. Dash, S. K. Sinhababu and G. N. Rao (R/R 436/85).

5. Alternative indirect volumetric method for determination of vanadium in Ferro-vanadium—M. R. Paramanik (R/R 437/85).

6. Warm Blast Cupola for small industries in rural areas—R. Sontok Singh and P. S. Virdhi (R/R 438/85).

7. Development of mini cupola for rural sector—R. Santok Singh, P. S. Virdhi and V. A. Altekar (R/R 439/85)

### Investigation Reports

1. Beneficiation of limestone from Bentibagda Area, from BSMDC, Ranchi—K. Visweswar Rao, P. D. Prasad Rao and N. Chakravorty (IR 1194/85).

2. Determination of some physical characteristics of sponge iron briquettes, produced at Orissa Sponge Iron from OSIL sponge iron fines—A. K. S. Mahapatra and K. N. Gupta (IR 1195/85).

3. Beneficiation studies on low grade phosphate from Beldih Mines, Purulia of M/s. W.B.M.D.T.C.—T. C. De, S. Sivaiah and N. Chakravorty (IR 1196/85).

4. Bench scale beneficiation studies on a sample of limestone from Bhawanathpur Mines from M/s. Bokaro Steel Ltd.—S. Sivaiah, M. V. Ranganathan, D. M. Chakravorty and N. Chakravorty (IR 1197/85).

5. Physical, Chemical, Mineralogical and Calcination studies on a limestone sample from M/s. Tamil Nadu Newsprint and Papers Ltd., Madras—T. V. K. Das, P. V. Viswanathan, P. R. Khangaonkar (IR 1198/85).

6. Pelletisation studies on iron ore concentrate from Tiruvaunamelai, Tamil Nadu—T. V. K. Das, K. Vijayaraghavan, P. V. Viswanathan, V. Mohan, C. Sankaran and P. R. Khangaonkar (IR 1199/85).

7. Determination of some physical characteristics of Sponge Iron and sponge iron briquettes produced at Orissa Sponge Iron Ltd., from OSIL sponge iron fines—M. C. Goswami and K. N. Gupta (IR 1200/85).

8. Froth flotation studies on Baragolai Coal Sample (60' Seam) from N.E.C., Assam—S. K. Sil, M. V. Ranganathan and N. Chakravorty (IR 1201/85).

9. Mineralogical and Locking study of Tailing sample from Sargipate Mine Project received from M/s. Hindustan Zinc Limited—S. Mohan Rao, B. Banerjee, A. Peravadhanulu and N. Chakravorty (IR 1202/85).

10. Beneficiation studies on the Salem Magnesite Samples sponsored by Mineral Development Board, New Delhi, bench scale and pilot plant tests part I, II and III:

    Part I—one Report.
    Part II & III—one Report.
    NML Unit, CSIR Complex, Madras (IR 1203/85).

11. Evaluation of imported boiler quality Tube for DCP ($2\frac{1}{2}$ Cr-1 Mo Steel) by SAIL, Ranchi—R. Prasad, R. Singh, T. R. Soni and Y. N. Tiwari (IR 1204/85).

12. Investigation on Stress rupture properties of $2\frac{1}{2}$ Cr-1Mo Steel produced by Alloy Steel Plant, Durgapur, W.B.—All staff of Creep laboratory (IR 1205/85).

13. Investigation on stress rupture properties of $1\frac{1}{4}$ Cr-$\frac{1}{2}$ Mo Steel produced by Alloy Steel Plant, Durgapur, W.B.—All staff of Creep laboratory (IR 1206/85).

14. Beneficiation studies of Silicons Manganese Ore received from M/s. Aditya Mineral, Nagpur, Maharashtra—P. N. Pathak, J. P. Srivastava and N. Chakravorty (IR 1207/85).

15. Investigation on convective superheater tube of coil No. 57. Tube No. 1, Unit No. 3 of 50 MW power station, MPEB, Korba—R. Singh, K. Prasad and R. Kumar (IR 1208/85).

16. Agglomeration studies on Iron Ore samples from Bailadila Deposits of M/s. MMDC, Hyderabad—S. C. Maulik, S. R. Ghosh, K. Visweswar Rao, D. M. Chakravorty, V. S. Chattoraj and N. Chakravorty (IR 1209/85).

17. Development of Magnesite briquettes and its ultimate utilisation of Magnesite dust sample from Almorah Magnesite—K. C. Roy, B. K. Mitra, P. C. Sen and M. R. K. Rao (IR 1210/85).

18. Studies on briquetting of lime produced from a sample of Calcite from M/s. Ind. Chemicals (IR 1211/85).

19. Froth flotation studies on Coal sample (20' Seam) from Baragolai Mines, Assam for M/s. CEMPDIL, Ranchi—S. K. Sil, M. V. Ranganathan and N. Chakravorty (IR 1212/85).

20. Investigation report on tests conducted on graphite ore from DORR-Rake Classifier and flotation cells to be used for graphite beneficiation project of TAMIN—P. D. Prasad Rao, K. Visweswar Rao, D. M. Chakravorty and N. Chakravorty (IR 1213 /85).

21. Beneficiation characteristics of Non-coking Coal Sample from Talcher Coal Field for reduction of Ash Cement and Alpha-Quartz Content. Phase I—Bench scale studies: Part I—heavy liquid separation study—P. D. Prasad Rao, K. Visweswar Rao, D M. Chakravorty and N. Chakravorty (IR 1214/85).

22. Beneficiation studies on a magnetite sample from SEMRA Mines of M/s. BSMDC Ltd.—J. P. Srivastava, K. Visweswar Rao, D. M. Chakravorty and N. Chakravorty (IR 1215/85).

23. Beneficiation studies on a magnetite sample from Salatua for BSMDC Ltd.—J. P. Srivastava, K. Visweswar Rao, D. M. Charavorty and N. Chakravorty (IR 1216/86).

24. Bench scale beneficiation studies on the low grade graphite samples from (1) Sewadih, (2) Manasote (3) Bishrampur Mines from M/s. BSMDC, Ranchi—P. N. Pathak, S. K. Sen Gupta, M. V. Ranganathan, D. M. Chakravorty and N. Chakravorty (IR 1217/86).

25. Beneficiation characteristics of Non-coking Coal sample from Talcher Coal Field for reduction of Ash content and $\alpha$-quartz content—S. Sivaiah, S. K. Sil, S. C. Maulik and N. Chakravorty (IR 1218/86).

26. Beneficiation of low grade chromite ore from Saurabil Mines, Orissa—R. K. Kunwar, H. Pathak and N. Chakravorty (IR 1219/86).

27. Beneficiation studies on overburden chromite samples from Saurabil Mines, Orissa—R. K. Kunwar, H. Pathak, D. M. Chakravorty and N. Chakravorty (IR 1220/86).

**Survey Reports**

1. Magnetite concentration cum grinding plant and graphite beneficiation plant of BSMDC, Ranchi.

2. Energy conservation techniques in fuel fired furnaces—A. K. Bose, P. C. Sen and M. R. K. Rao.

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